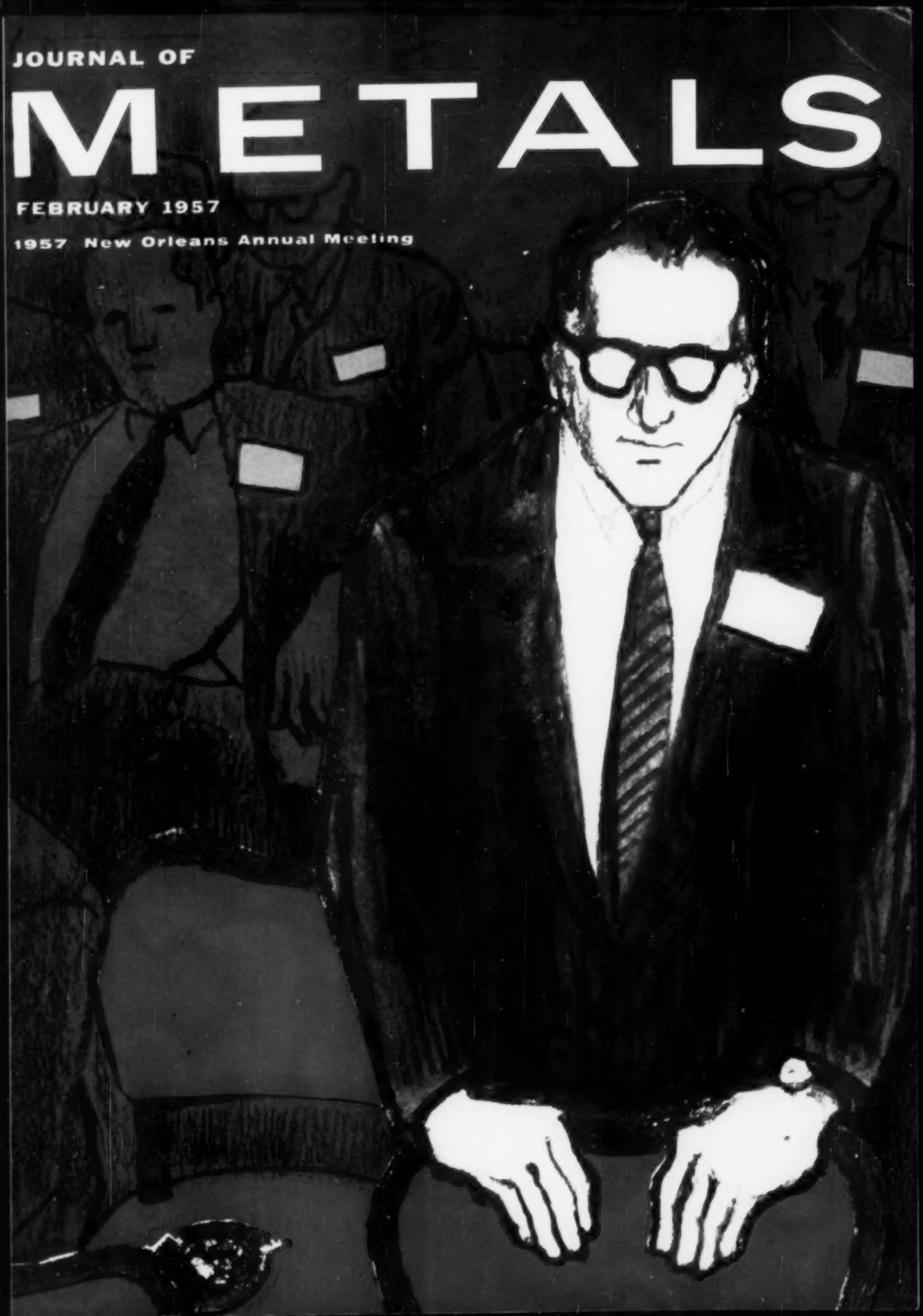
JOURNAL OF

# METALS

FEBRUARY 1957

1957 New Orleans Annual Meeting



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| Carbon | 0.10% max. | 0.10% max. | 0.10% max. |
| Size | 2 in. x Down | 2 in. x Down | 2 in. x Down |

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| | |
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|---|---|
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| Iron | 2.00% max. | Iron | 1.00% max. |
| | | Carbon | 0.15% max. |

Crushed Size: ¼ in. x Down

JOURNAL OF

# METALS

VOL. 9, NO. 2

FEBRUARY 1957

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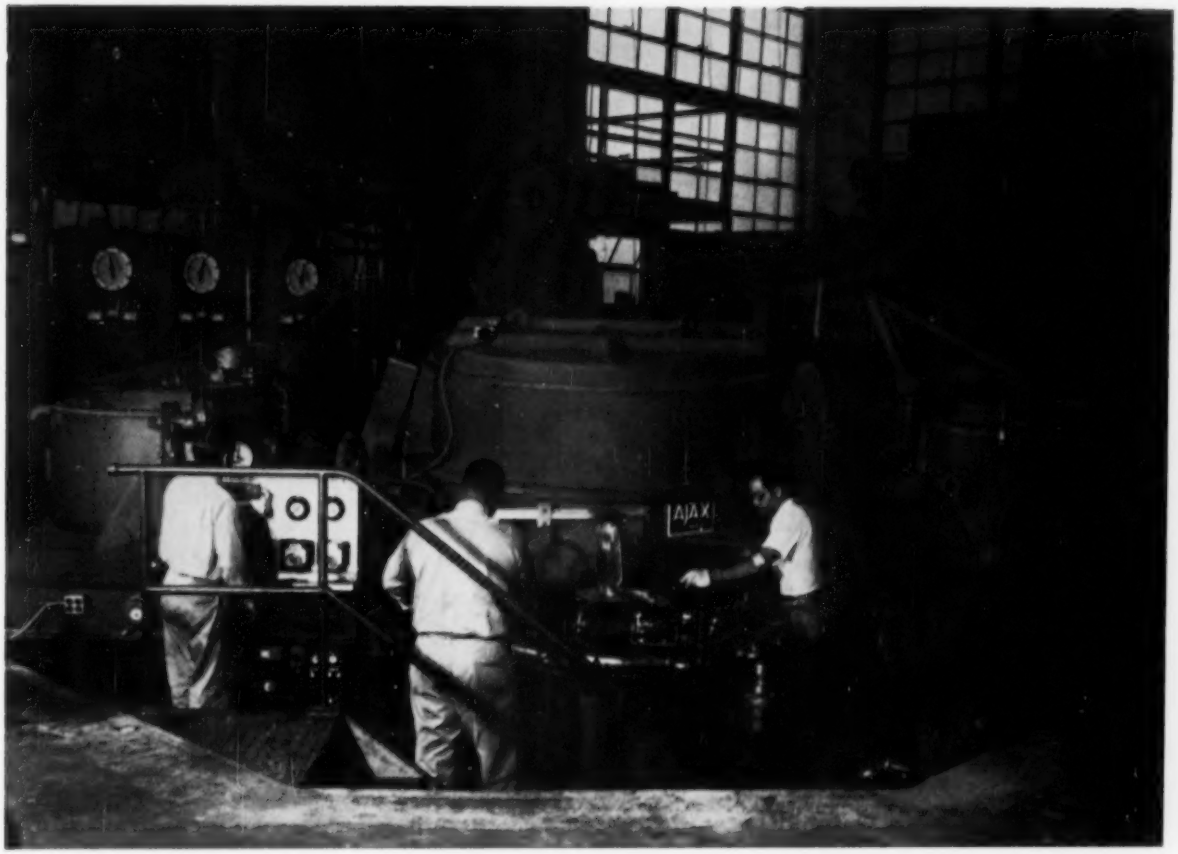


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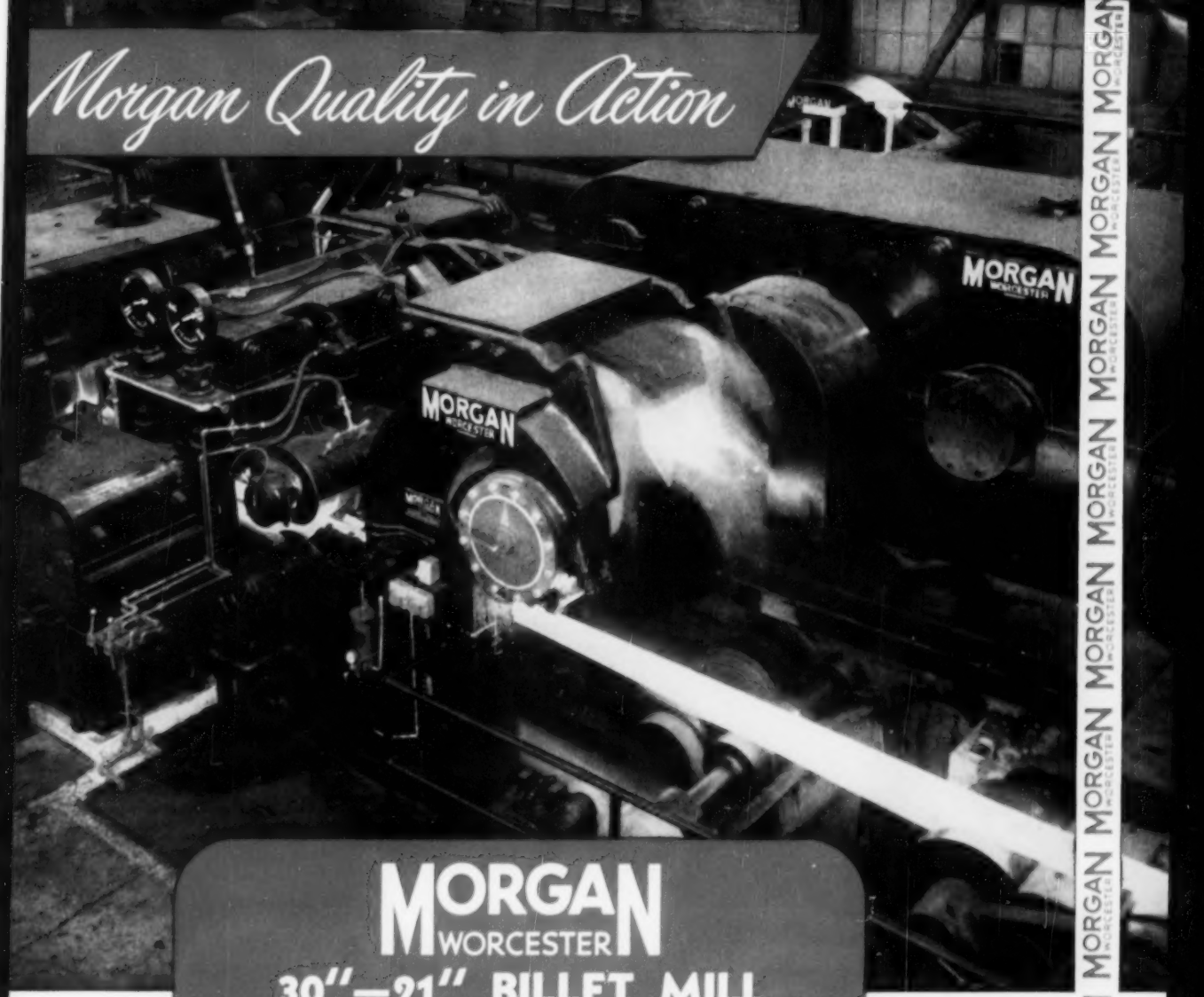


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## The Year 1956

### . . . Lead-Zinc Production Rises as Consumption Declines

During 1956, U. S. production of slab zinc amounted to a record 1,063,000 tons, an increase of some 3 pct over 1955. Zinc consumption from 1955 to 1956 dropped by some 150,000 tons, reflecting lower automobile output, the steel strike, and reduced purchases by the brass industry. Lead output was up about 4 pct, but consumption was less adversely affected than zinc. In both cases price stability was maintained by U. S. government purchases for military stockpile.

### . . . Copper Supplies Meet Demand

U. S. production of crude copper reached 1,270,000 tons in 1956, more than 100,000 tons above 1955 output and the highest on record! Industrial consumption increased, but because copper prices were high throughout most of the year, the Government did not enter into new stockpile commitments. Expansion is planned by the industry, and mine capacity of the non-communist world should reach almost 4 million tons by 1962 compared with some 3 million tons today.

### . . . Nickel Output at New High

International Nickel reports 1956 nickel production of non-communist world reached almost 450 million lb, compared with 427 million in 1955. Estimated deliveries by principal producers are: Inco 285 million lb; Falconbridge 43 million lb; Sherritt Gordon 19 million lb; U. S. Government's Nicaro, Cuba plant 31 million lb, Hanna Nickel Smelting in Oregon 14 million lb, and the French Société "Le Nickel" 22 million lb. Some 65 pct of this nickel was consumed in the U. S. Increased amounts went to defense, although the quantity available for civilian applications was somewhat greater than 1955.

### . . . Aluminum Capacity Now Equals Current Demand

The 1956 production of primary aluminum in the U. S. reached 1,680,000 tons, some 7 pct above 1955 and the fifth successive year that the industry has set a new production record. During the year primary producers increased their annual capacity by more than 125,000 lb, bringing total U. S. capacity up to 1,760,000 lb by the year's end. Production of aluminum ingot from scrap also continued at a high level.

### . . . Steel Production Second Highest On Record

Despite the loss of an estimated 11 million tons during the strike, the steel industry of America achieved a production of 115.2 million tons of ingots. During the year, the industry increased capacity by 5 million tons, bringing total ingot capacity of 133.5 million tons, corresponding to a blast furnace capacity of 86.8 million tons. To meet this expansion, the industry spent an estimated $1200 million during the year. . . . Steel production of the European Coal and Steel Community reached a record level, 62.4 million tons, an increase of almost 8 pct over the previous year. In the United Kingdom, steel production reached a record 23.1 million tons in spite of labor disputes and the economic dislocation caused by Suez.

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**Chemical Engineering Practice,** Vol. II. Solid State, ed. by H. W. Cremer, *Academic Press Inc.*, 632 pp., $17.50, 1956—This is a collection of 15 papers by British authors on various aspects of matter in the solid state. Discussions of powder metallurgy, corrosion of metals, porous masses, and metallic alloys are included.

**Induction Heating Practice,** by D. Warburton-Brown, *Philosophical Library*, 189 pp., $10.00, 1956—This is a practical handbook on the application to industrial problems of induction heating, with special reference to its use in brazing, soldering, annealing, hardening, and tempering.

**Progress in Semiconductors, Vol I,** ed. by A. F. Gibson, P. Aigrain, and R. E. Burgess, *John Wiley & Sons Inc.*, 217 pp., $8.00, 1956—This is the first of a series intended to present critical reviews of the literature, as well as original work, in the field of semiconductors and allied subjects. Subjects considered in the present work are: silicon, germanium filament, high-frequency transistors, and photomagnetoelectric effect.

**Molybdenum,** by L. Northcott, *Academic Press Inc.*, 222 pp., $6.80, 1956.—This book, No. 5 in the Metallurgy of The Rarer Metals Series, is intended to be both text and reference of available information on molybdenum, giving special attention to low oxidation resistance and embrittlement at low temperatures.

**Phase Diagrams in Metallurgy,** by F. N. Rhines, *McGraw-Hill Book Co. Inc.*, 311 pp., $12.00, 1956.—This text is aimed at undergraduate engineering students whose primary interest is in the application of phase diagrams to metallurgical problems. Therefore, the approach is from the viewpoint of their structure and use, rather than their determination and the emphasis is on the types of equilibria of special interest to metallurgists.

**Industrial Engineering Handbook,** ed. by H. B. Maynard, *McGraw-Hill Book Co. Inc.*, 1504 pp., $17.50, 1956.—This is an illustrated reference and guide for the practicing engineer and business manager on the principles, data, methods, and procedures of the whole field of industrial engineering.

**Please Order These Publications from the Publishers**

**The Physics of Nuclear Reactors,** British Journal of Applied Physics Supplement No. 5, *The Institute of Physics*, 47 Belgrave Square, London, S. W. 1, Eng., 112 pp., $3.60, 1956.—This volume presents papers on problems of nuclear reactors given at the time of the British Nuclear Energy Conference held in London, July 1956.

**Symposium on Powder Metallurgy,** Special Report No. 58, *The Iron and Steel Institute*, 4 Grosvenor Garden, London, S. W. 1, Eng., 390 pp., $9.50, 1956.—This volume contains 50 papers presented in four groups: manufacture, properties, and testing of powders; principles and control of compacting and sintering; manufacture and properties of structural engineering components; and powder metallurgy of high melting-point materials.

**Théorie et Technique de la Radiocristallographie,** by A. Guinier, *Dunod*, 92 Rue Bonaparte, Paris 6e, 750 pp., 9500 francs (app. $23.50), 1956.—This work presents the latest progress achieved in the technique of X-rays and the interpretation of diagrams. Stress is placed on all the applications of X-rays in the laboratories of chemistry, solid physics, and metallurgy, and an important chapter is devoted to crystalline imperfections, so important to the physics of metals.


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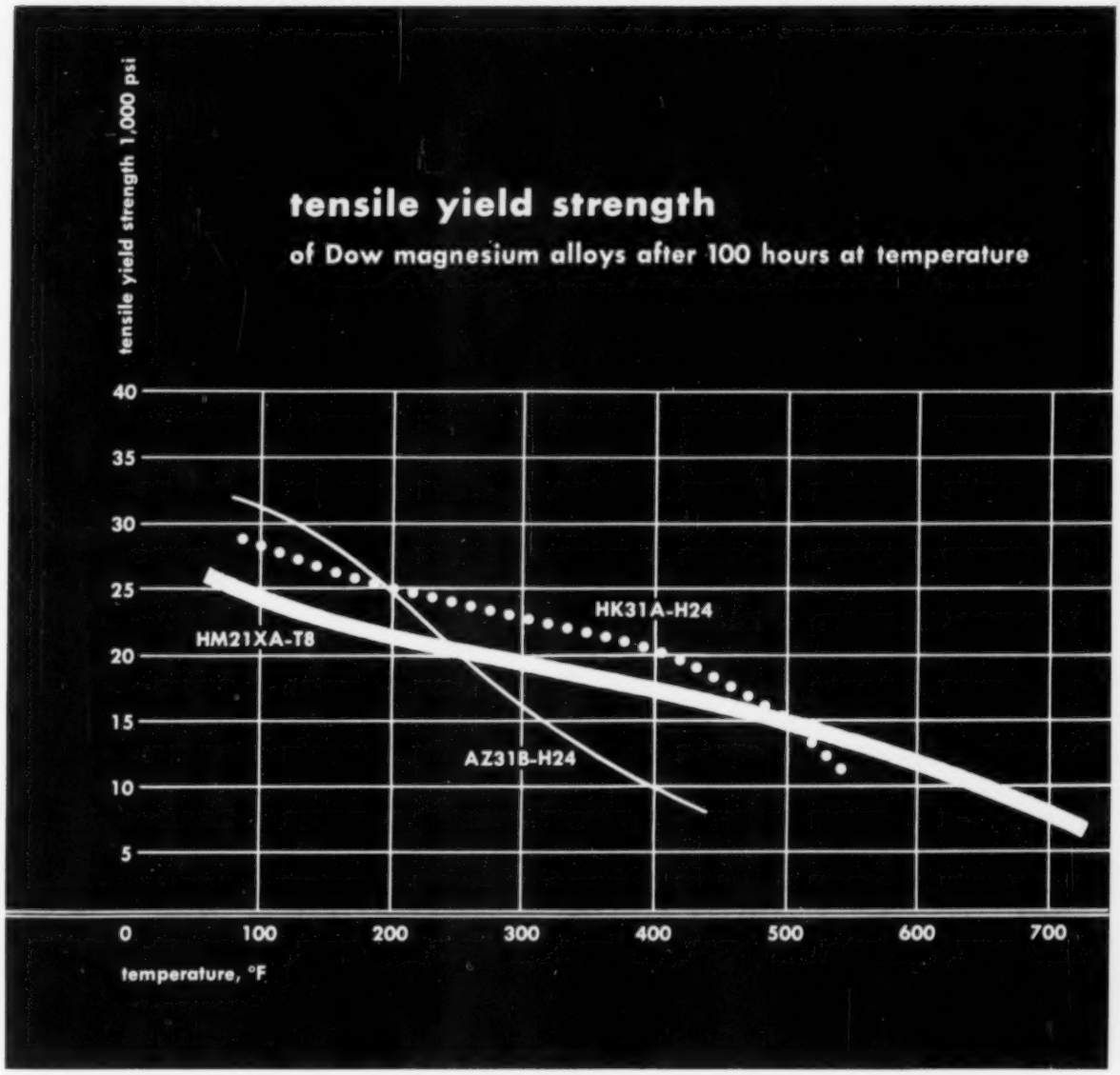


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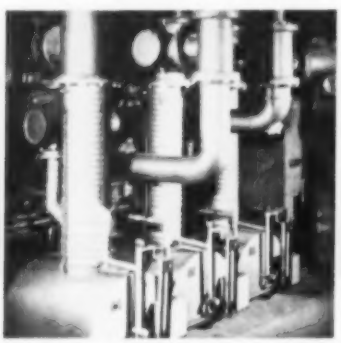

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# U.S.I. CHEMICAL NEWS

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## *Greenleaf to Handle Market Development of Zirconium And Titanium for U.S.I.*

William C. Greenleaf has been named Manager of Metals Development for U.S.I. and will be responsible for market development and sales of zirconium and titanium sponge, to be produced from U.S.I.'s two new plants now being built in Ashtabula, Ohio.

A recognized authority in the field of zirconium and titanium, Mr. Greenleaf presented three papers during the 1955 Atomic Industrial Forum Zirconium Program, describing (1) melting of zirconium, (2) fabrication of zirconium, and (3) zirconium mill products and prices. He also conceived and developed processes now in general use for producing both wide-sheet and flat bar titanium and zirconium by continuous strip-mill techniques.

## Faye Named Manager of Sodium Product Sales

Martin Faye has been named Manager of Sodium Product Sales for U.S.I. and will be responsible for coordinating sales and sales promotional activities for metallic sodium, "U.S.I. Esterbactic" acid and related products.

Mr. Faye joined the U.S.I.-National Distillers organization in 1952 in the Market Research and Development Department.

TECHNICAL DEVELOPMENTS

*Information about manufacturers of these items may be obtained by writing the Editor, U.S.I. Chemical News*

**A zirconium-copper alloy** now available is reported to combine high electrical conductivity with good strength retention at elevated temperatures. Suggested for electrical motor commutators serving above 500°F. where strength is needed. Conductivity is 95.6% of copper. **No. 1193**

**Titanium wire cloth** is now on the market in sizes from 60 mesh to coarser spacing. It is reported to be particularly suitable for filtering or screening highly corrosive materials. **No. 1197**

# Zirconium and Titanium Lick Roughest Corrosion Problems

### Metals to be Available at Lower Cost in Future; Provide Long-Lived Materials of Construction; Complement Each Other on Corrosion Resistance

It is now practical to fabricate equipment which is *corrosion-resistant to almost every substance encountered in industry*, by using either zirconium or titanium metal. Alloys of either metal, or possibly both, may extend the range even further.

Industrial applications of these metals have been hampered by two considerations: availability and price. Zirconium will shortly be produced at a rate sufficient to supply industrial needs, and titanium has been available to industry during the past two years. And it is expected that increased production of these metals during the next few years will result in significant price decreases.

Twelve to thirteen thousand tons of titanium were available in 1956 and present construction schedules indicate that the figure will rise substantially in 1957. U.S.I.'s new plant alone will add 5,000 tons to the annual production capacity by the end of the year. Zirconium will be on the market in quantity by mid-1957. When its new plant at Ashtabula, Ohio gets under way, U.S.I. will be able to supply 500,000 pounds or more per year to commercial users in addition to its Atomic Energy Commission commitments of 1,000,000 pounds per year.

#### New Metals Economical

Chemical process equipment can now be fabricated from titanium for a little over twice the price of stainless steel, and it is expected that in the future titanium equipment will be only 50-75% higher than stainless. Commercial grade zirconium equipment will probably be priced only 75-100% higher than stainless when volume production is reached.

### Typical Corrosion Resistances* of Zirconium and Titanium

| Corrosive Media | Metal Resistance | |
|---|---|---|
| | Zirconium | Titanium |
| Sulfuric Acid | excellent to good below 80% | good below 5% |
| Nitric Acid | excellent | excellent |
| Hydrochloric Acid | excellent | good below 10% |
| Phosphoric Acid | excellent to fair below 85% | poor |
| Chromic Acid | excellent | excellent to good |
| Aqua Regia | poor | excellent |
| Wet Chlorine Gas | poor | excellent |
| Chlorine Water | excellent | excellent |
| Sodium Hydroxide | good below 90% | good below 50% |
| Ferric Chloride | poor | excellent |
| Calcium Chloride | excellent | excellent |
| Cupric Chloride | poor | excellent |
| Sodium Chloride | excellent | excellent |
| Ammonium Chloride | excellent | excellent |
| Aluminum Chloride | excellent | excellent to fair |

*Above data do not cover all conditions because of space limitations. Literature references may be obtained by writing the Editor, U.S.I. Chemical News, 99 Park Ave., N. Y. 16, N. Y.

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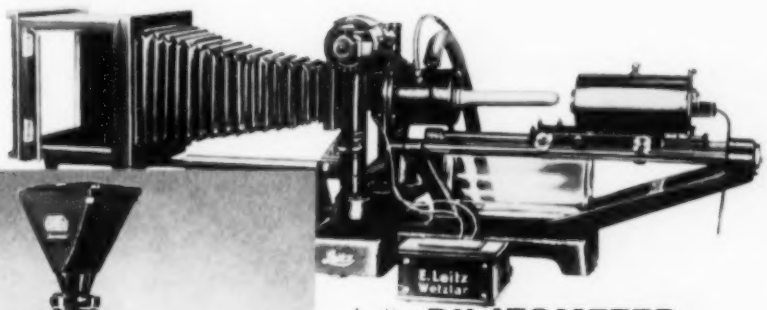

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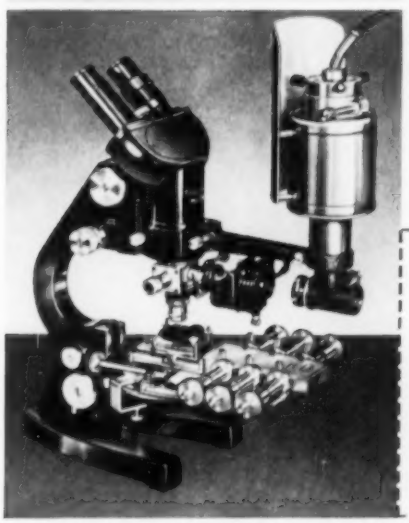

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# Upstream!

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The titanium industry also has been fighting upstream these past five years. A succession of production rapids and metallurgical waterfalls have been surmounted. The dangerous hydrogen whirlpool was successfully skirted. The industry has now moved through the less white headwaters to fully competitive equality with other structural metals.

Titanium alloys of high strength, light weight and outstanding corrosion resistance are available from T.M.C.A. in all mill forms and in a full range of sizes and gages — sheet, bar, billet, extrusions, tubing and wire. Special heat-treated sheet of very close gage and flatness tolerances is in production for advanced aircraft and missiles.

Further expansions of sponge production and metal finishing facilities at T.M.C.A. are bringing titanium within reach of an ever-expanding market. Technical information and specialized engineering services are available for solving those applications having a strength, weight or corrosion challenge.

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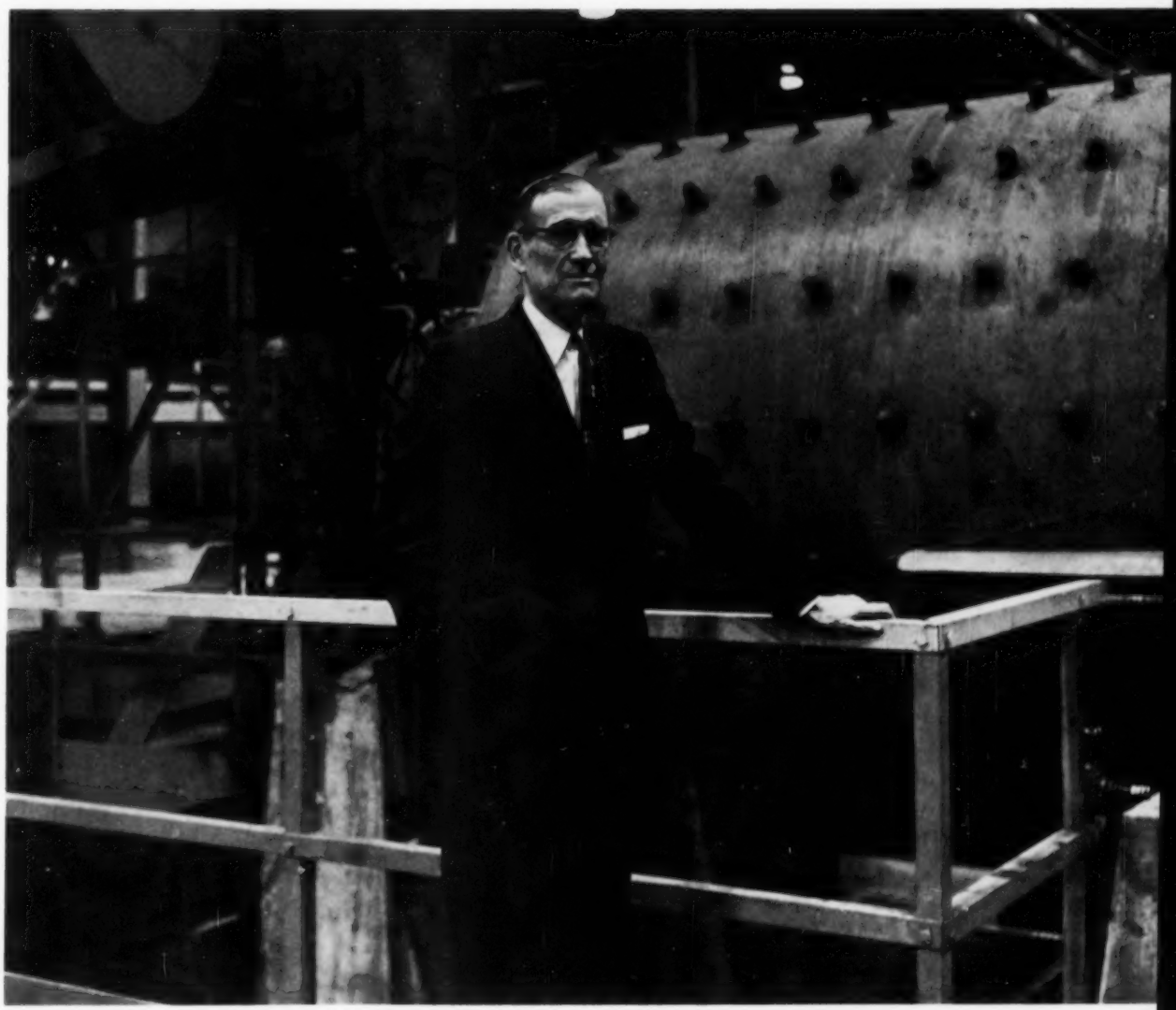


GROVER J. HOLT

*For a Profile of Grover J. Holt,*
*1957 AIME President, Turn the Page*

GROVER J. HOLT, member of the Institute since 1941, was born in Northwood, N. D., in 1894. After receiving a B.S. degree in mining engineering in 1916 from the University of North Dakota, he became a research fellow in metallurgy at the University of Utah and last year was awarded the honorary degree of Doctor of Engineering by the Michigan College of Mining & Technology.

His career has provided professional contact with a wide range of the mineral industry—from western nonferrous operations to all aspects of iron ore and its preparation for blast furnace use.

During World War I he served with the AEF as a Lieutenant, U. S. Army Engineers, and following this service spent two years in the Bunker Hill & Sullivan smelter and underground properties at Kellogg, Idaho.

In 1920, Mr. Holt joined U. S. Steel Corp. as a mining engineer and has made the Lake Superior iron range region his home area ever since. Here his activities have covered the full scope of mining and beneficiation in the areas of operation, research, and finally overall management.

He became assistant to the vice president of Butler Brothers in 1930 and during the next decade was active in pointing out depletion of reserves of direct shipping iron ore, and in developing methods to process lower grade reserves.

In 1940–1941 he designed the first sink-float installation using ferrosilicon media, which was placed in operation at Butler's Patrick plant, and also presented the first paper on the application of this process at an AIME session during that period.

One credo for the engineer says, "First be sure, then go ahead." The ability to determine the crucial point—when you are really sure—is the mark of a doer, not the dreamer. The doer must also have the facts. Mr. Holt has not only pioneered in iron ore research himself—he early recognized its importance to development of the remaining reserves of our basic raw materials for the making of steel.

They tell one story that perhaps best defines the difference between the dedicated researcher after knowledge for its own sake, and the engineer who must balance the search for the truth with the need for action. It seems that Grover Holt and one of his top technical people were riding across the Minnesota farm country when they passed a flock of sheep. Mr. Holt commented that the sheep had recently been closely sheared. The research man agreed—with qualifications—"At least they are on this side."

Mr. Holt joined the Cleveland-Cliffs Iron Co. as Chief Engineer in 1942, becoming Manager, Minnesota Mines, in 1950, and General Manager of Mines for Minnesota and Michigan in 1953. Here his role has been that of a builder. During this period CCI established a beneficiation laboratory, and from work done there and in the Engineering Dept., a succession of plants have sprung up to treat Minnesota and Michigan low grade ores.

In first seeing the problem: diminishing reserves of direct shipping ore, and then taking the steps to meet these problems, Grover Holt has an outstanding record of participation in first steps. During the 1930's he joined the few who spoke out at that time about the ore needs of the future; before World War II he delivered the first paper on the corner stone process of intermediate grade ore beneficiation: heavy media, now used almost universally on the range and elsewhere for coarse ore concentration.

In 1943 CCI was one of the first companies to build a new laboratory to tackle beneficiation problems. Also, more recently the CCI company in Michigan put into operation the first commercial iron ore flotation plant.

Foresight without the ability to plan ahead is of little value. It is perhaps significant CCI's plants of recent years have had ample and detailed testing, development, and planning behind them.

At a turning point in AIME development, Grover Holt comes well prepared to the task of the Presidency. Having played a major role in drafting the new By-laws, he brings a unique background to a unique opportunity. His will be the task of guiding the first and formative year of operation under a new pattern which he helped design.

For the future, ways must be found to take advantage of the flexibility of the three-society framework, while retaining the values of the stability and strength of the Institute. Foresight and ability to plan for the future will stand AIME in as good stead as it has the iron ore industry.

Mr. Holt lives in Ishpeming, Mich., where he *occasionally* has an opportunity to take advantage of the excellent local hunting and fishing facilities in that area.

R. A. Perkins and W. O. Binder

# IMPROVING HOT-DUCTILITY OF 310 STAINLESS

*Under some conditions of manufacture, austenitic stainless steels exhibit a tendency to hot-crack during fabrication. Hot-cracking has been observed in casting, heating for working, ingot breakdown, hot-rolling, tube-piercing, and other hot-forming operations. It has been particularly troublesome in both the weld and heat-affected zones of welded heavy sections. In view of the reduced yields and economic losses which result from this behavior, there is an important need for a better understanding of factors which control hot-cracking tendencies.*

LOW ductility of steel at elevated temperatures—hot-shortness—is by far the most important factor which promotes hot-cracking. Hot-shortness can result from contamination with such tramp elements as lead, silver, or tin. Careful selection of raw materials and scrap is an obvious solution in this case. But low hot-ductility also can result from the formation of an austenite-ferrite duplex structure, or from variables in melting practice which, at present, are not clearly defined. These variables are troublesome causes of hot-short failures and, in general, are more difficult to detect and overcome.

## In Duplex Structures

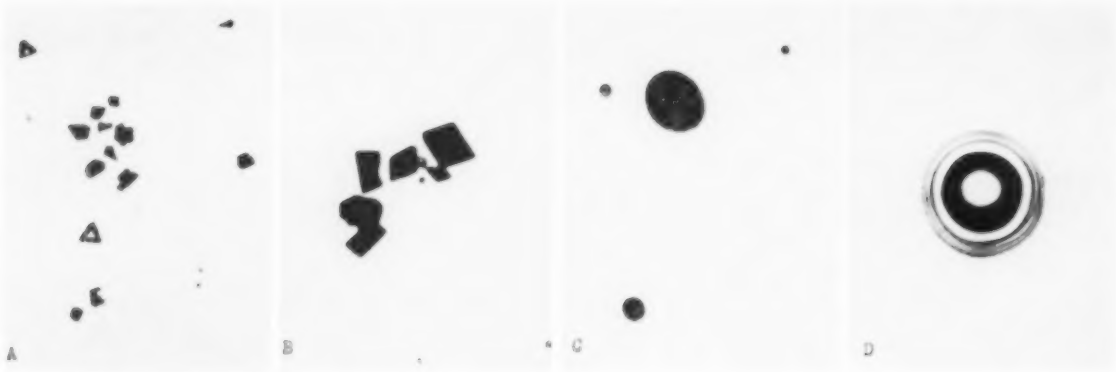
Hot-shortness associated with duplex structures has received considerable attention, and methods for control of this problem are fairly well defined. Bloom, Clarke, and Jennings[1] related hot-ductility to microstructure. They pointed out the detrimental effects of ferrite in austenitic steels and indicated the importance of maintaining a balanced composition. Post, Schoffstall, and Beaver[2] described the effect of ferrite and composition balance on rolled billet yields in hot-working types 308 and 316 stainless steels. The importance of the amount and distribution of ferrite in the structure in relation to hot-short behavior has been shown by Lula and Henke.[3] Small amounts of ferrite are not necessarily detrimental; however, large amounts, particularly in a network distribution, can cause serious hot-cracking during working. Generally, this type of hot-shortness can be avoided by proper balance of composition and soaking-pit practice.

R. A. PERKINS and W. O. BINDER are Research Metallurgist, Metals Research Laboratories, and Assistant Manager, Product and Process Development Dept., respectively, Electro Metallurgical Co., Div. of Union Carbide and Carbon Corp., Niagara Falls, N. Y.
Paper was presented at the Electric Furnace Steel Conference, Chicago, December 1956.

## In Single-Phase Structures

A much more troublesome type of hot-shortness is that which occurs in single-phase austenitic steels. Although low hot-ductility appears to be characteristic of these highly alloyed structures, in many compositions it can be overcome by changes in melting practice. Consequently, incidence of this behavior will vary from heat to heat within a given grade as well as for the different grades of austenitic steels, depending on conditions of manufacture. The problem has been studied extensively, but causes and methods for control are not clearly defined.

Melting practice, and deoxidation in particular, controls many of the metallurgical factors which are believed to contribute to hot-shortness in austenitic steels. The importance of deoxidation and its effect on cracking tendencies has been described by several investigators. Hamilton[4] correlated tube-piercing behavior and oxygen content. Newell[5,6] and Speranski[7] studied effects of silicon, titanium, and



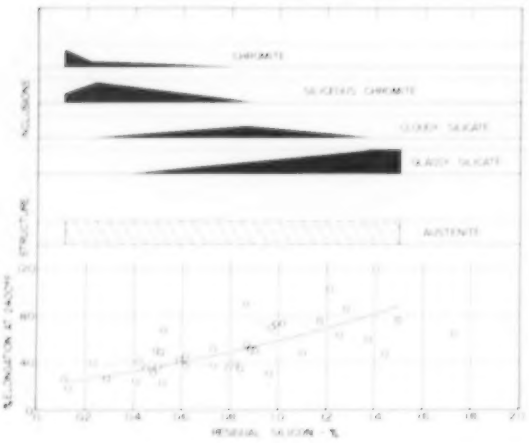



Fig. 1—SILICON. Hot ductility of steels increased gradually with increasing silicon content. Although a considerable scatter in results was observed, the trend was clear. Changes in reduction of area paralleled changes in elongation; samples with high elongation values generally pulled to a point, showing better than 99 pct reduction. Low ductility samples contained numerous checks and fissures over the entire gage length. Ferrite was not detected in either the cast or wrought structure in steels containing up to 1.73 pct Si.

Type and relative distribution of the inclusions changed markedly with increasing silicon, as shown at the left. These are pictured above: A, idiomorphic crystals of Chromite, which tended to segregate in small clusters, X500; B, Siliceous Chromite, noted by increased transparency and loss of sharp angular form, X2000; C, Cloudy Silicate, which is spherical, X2000; and D, the familiar Glassy Silicate, X2000. Increasing silicon from 0.1 to 0.2 pct residual, reduced the total oxygen content, but additional silicon had little effect. Hot ductility could not be correlated with oxygen content.

other deoxidizers. Although their results in some cases were contradictory, a general beneficial effect of deoxidizers was observed. More recently, improvements in hot-ductility and working properties of high-alloy steels as a result of rare-earth additions have been found.[7] Although the importance of deoxidizers has been recognized, no systematic study of the various deoxidizing elements has been made generalizing on this behavior.

## Present Study Shows Effects of Additions of Deoxidizing Elements

Eight deoxidizing elements were added to type 310 stainless steel. Silicon, manganese, vanadium, aluminum, titanium, zirconium, cerium (rare earths), and calcium were selected for the tests. Complex deoxidation practices purposely were omitted to simplify interpretation of results. Type 310 steel was selected as a base for the experiments, since its hot-ductility is known to be sensitive to melting practice. Short-time tensile tests at 2400 F., a temperature at which type 310 steel exhibits low ductility, were used as measure of relative ductility. Initially, tests were conducted on hot-worked specimens to minimize effects of chemical segregation and variations in grain size. The investigation later was broadened to include studies of ingot metal.

The results of this study cannot be applied directly to commercial practice, since changes from the test conditions probably will alter the relationships found to some extent. However, the results are believed to have general broad applicability, particularly to the behavior of hot-worked austenitic steels.

## Experimental Procedure

Melting and pouring practices, chemical composition, and hot-working and testing procedures were maintained constant in all experiments so that observed effects could be related directly to the deoxidation additions. Twelve-pound heats, having a nominal base composition of 25 pct Cr, 20 pct Ni, 0.03 pct C, 0.04 pct N, 0.01 pct S, 0.01 pct P, and balance iron, were induction-melted without slag cover. Silicon and manganese were held at 0.20 to 0.70 pct and 1.0 to 1.5 pct respectively, except in those heats where the effects of these elements specifically were studied. Other special deoxidation additions, as summarized in Table I, were added directly to the crucible immediately before the heats were tapped. All heats were poured into cold, 2-in. sq, cast-iron, ingot molds.

Cone forgeability,[8] hot-twist,[9,10] and hot-tensile[11] tests were used to measure hot-ductility in preliminary experiments. Of these, the short-time tensile test was selected for subsequent studies, primarily on the basis of convenience and of providing the quantitative measure of ductility which was desired. Elongation in 1 in. and reduction in area of specimens loaded to failure were taken as a measure of ductility. Initially, samples were prepared from hot-worked bar to minimize effects of chemical segregation and variable grain size. Subsequently, tests were made on specimens machined directly from the ingot to compare effects in cast metal with those observed in wrought metal.

Standard ASTM 0.25-in. diam specimens were prepared from hot-worked 0.5-in. diam bar, and

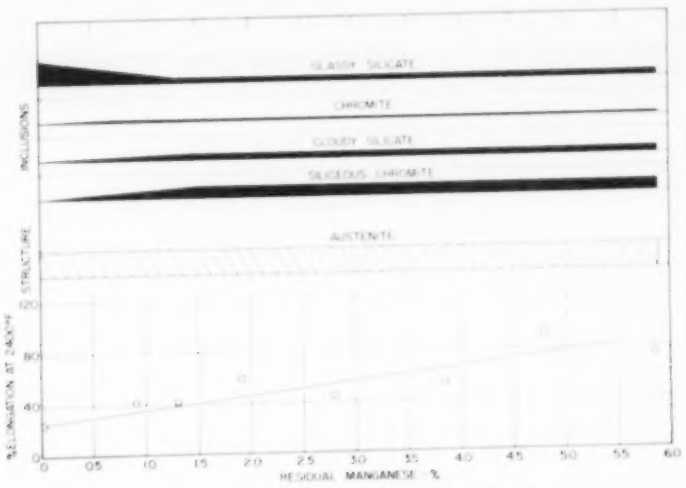
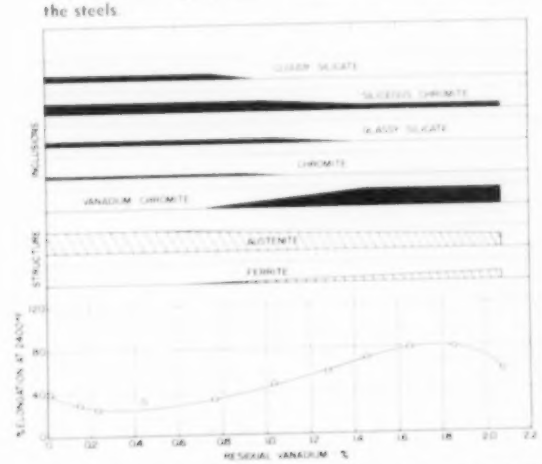


Fig. 2—MANGANESE. Behavior of manganese from nil to 5.84 pct was investigated in steels containing 0.5 to 0.6 pct Si. Hot ductility increased gradually with increasing manganese content from a minimum of 25 pct elongation at nil Mn to 85 pct at 5.2 pct Mn. Addition of 4.25 pct Mn resulted in an average elongation of 70 pct at 2400°F. Ferrite was not detected in any of the steels. The micrograph shows inclusions at 4.77 pct Mn, X250. Manganese had no significant effect on total oxygen content of the steels.



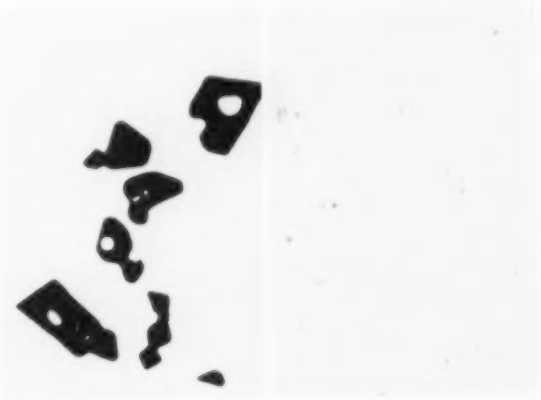



Fig. 3—VANADIUM. Addition of vanadium in excess of about 0.7 pct produced a significant change in hot ductility and altered the type of inclusions. Ferrite was observed in both the ingot structure and in the wrought bar, and its first appearance coincided with an increase in ductility. At approximately the same composition, a different type of inclusion appeared, in which vanadium is substituted for part of the iron or chromium. This Vanadium Chromite is shown on the left at X2000, and the micrograph at right, containing 1.65 pct V, shows the distribution. There was no significant change in oxygen content with vanadium addition.

0.505-in. diam specimens were machined from a longitudinal quarter section of the cast ingots. Two samples of each were tested at 2400°F at a constant crosshead speed of 0.04 in. per min. The samples were homogenized 15 min at temperature and then loaded to failure. Tests were made in an atmosphere of flowing argon to minimize surface oxidation; however, minor leaks in the system resulted in the formation of a thin, adherent, oxide scale.

Identification of inclusion types was based solely on visual observation, using data from the *Metals Handbook*[10] and Portevin and Castro[11] as a guide. No attempt was made to analyze the inclusions for constitution and structure. The system of nomenclature used to describe inclusions should not be interpreted to represent actual compositions. It is believed that over the wide range of alloy compositions studied, significant differences in chemistry exist between inclusions of similar outward appearance.

Microstructure was determined by metallographic examination of samples water-quenched from 2400°F. Steels also were tested with a magnetic balance to determine relative changes in the amount of delta ferrite in the structure.

The wrought bar was analyzed for oxygen, using vacuum-fusion techniques and for residual silicon and special deoxidizer.

## Results with Hot-Worked Steels

Individual results for each element added are shown in the accompanying charts and micrographs.

Results, as summarized in Table III, show that all eight elements could significantly improve the tensile ductility of the steel at 2400°F in the hot-worked condition. Ductility was dependent on the residual amount of the deoxidizer, except for calcium which was not studied on the basis of increased residuals. The ductility of steels treated with vanadium, aluminum, titanium, and zirconium passed through a maximum with increased additions. Decreased ductility at high residuals may be associated with the forma-

*(text continued on p. 244)*

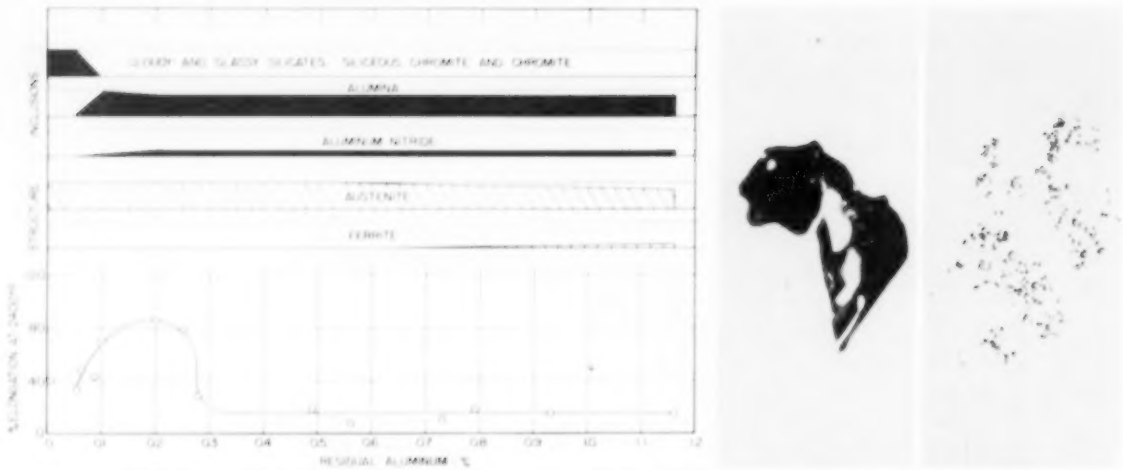



Fig. 4—ALUMINUM: Effect of aluminum on hot ductility was very marked, major changes occurring at low residuals and in a narrow range of composition. Aluminum contents in excess of 0.6 pct promoted the formation of ferrite in the microstructure. No change in tensile ductility was observed with the appearance of ferrite. However, above 1.2 pct Al, specimens cracked severely during forging. Inclusions were mainly of the alumina type, shown above left, at X2000. These showed a strong tendency to segregate in clouds, above right, which has 0.49 pct Al at X100. Above 0.10 pct residual Al, total oxygen was reduced from the normal 0.03 to 0.05 pct level to less than 0.01 pct.

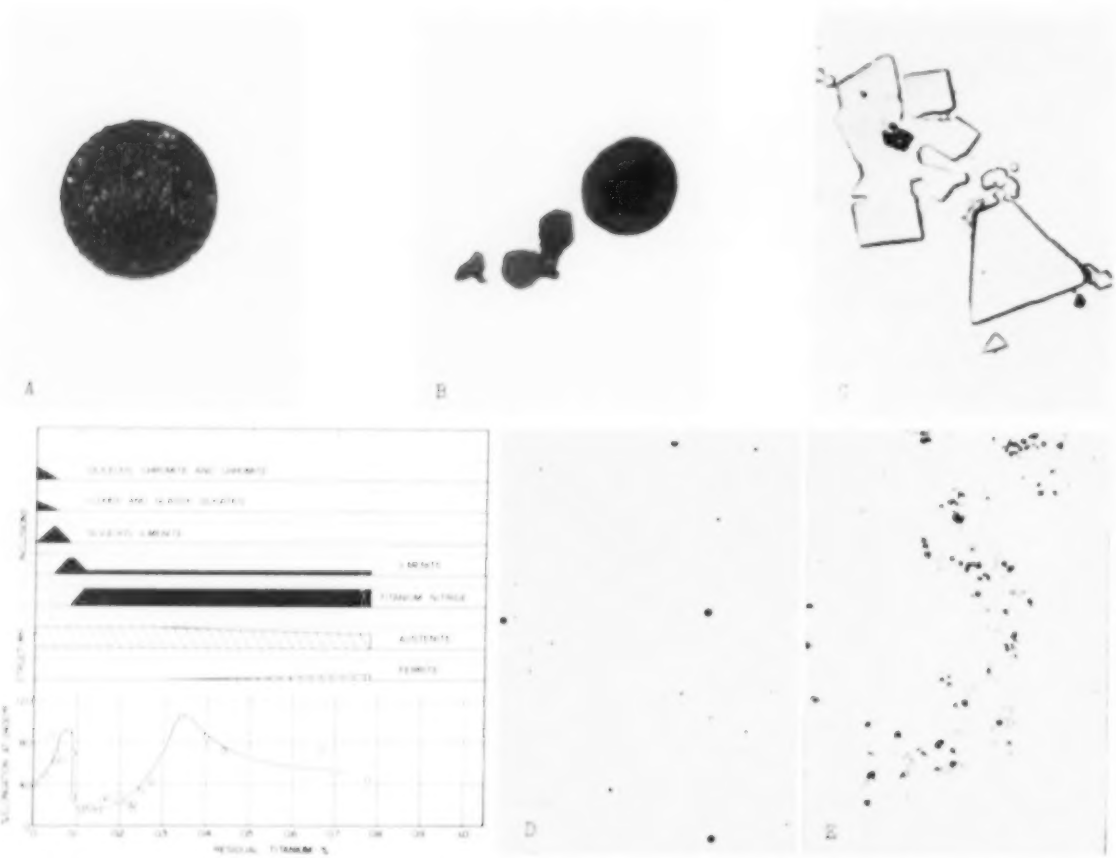



Fig. 5—TITANIUM: Two ranges for optimum ductility of 70 pct elongation existed: 0.05 to 0.08 pct Ti and 0.30 to 0.47 pct Ti. The best combination of ductility and cleanliness was provided in the lower of the two ranges. Ferrite was observed above 0.3 pct Ti and became abundant with increasing Ti content in both cast and wrought samples. Titanium altered the types of inclusions formed, as shown above. Picture A shows a duplex silicate-ilmenite, similar to cloudy silicates, but with precipitated ilmenite rather than chromite, X2000. Above 0.08 pct Ti, ilmenite (B) and titanium nitride (C) inclusions replaced the siliceous ilmenite, both shown at X2000. The nitrides segregated into large clusters, as shown in E (0.22 pct Ti, X250). In nitride-free samples (D 0.09 pct Ti, X250) inclusions were random. Above 0.10 pct residual Ti, oxygen was reduced to 0.01 to 0.03 pct.

Fig. 6—ZIRCONIUM. Three zirconium alloys were used, as noted in Table I; these did not significantly alter the results in the range tested. It can be seen that changes in hot ductility coincided with the occurrence of oxide inclusions (most likely $SiO_2$-$ZrO_2$), shown in A at X2000, and the nitride inclusions, shown in B at X2000. Ductility decreased as ferrite appeared. The oxides showed a slight tendency to segregate into small clusters, as shown in C with 0.04 pct Zr at X250; and the nitrides had a marked tendency to segregate into large clusters, shown in D with 0.41 pct Zr at X250. At 0.025 pct residual Zr (0.10 pct added), oxygen content was reduced to a 0.01 to 0.03 pct level.

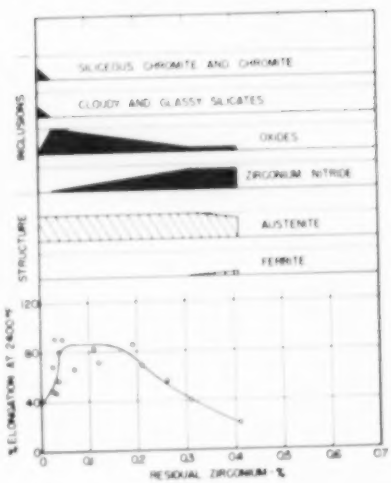


Table I. Deoxidation Additions

| Deoxidizer | Alloy | Particle Size | Method of Addition |
|---|---|---|---|
| Mn | Metallic Mn | ½ in. x down | Surface |
| V | High-Purity Metallic V | ¼ in. x down | Surface |
| Si | Metallic Si | ¼ in. x down | Surface |
| Al | 2S Al Rod | ¼ x ½ in. | Surface |
| Ce | Lan-Cer-Amp | ½ in. x down | Submerged |
| | Hi-Lanth | ½ in. x down | Submerged |
| Ti | 30 pct Grade Ferrotitanium | ½ in. x down | Surface |
| Zr | 75 pct Ni, 25 pct Zr (special) | ½ in. x down | Surface |
| | 12-15 pct Zr, 39-43 pct Si, Fe | ½ in. x down | Surface |
| | 35-40 pct Zr, 47-52 pct Si, Fe | ½ in. x down | Surface |
| Ca | Ca-Si | ¼ in. x down | Submerged |

Table II. Hot Ductility of Cast Versus Hot Worked Metal

| Heat No. | Deoxidizers, Pct Si | Mn | Al | Ti | Elongation in 2 In. at 2400°F, Pct As Cast | Hot Worked |
|---|---|---|---|---|---|---|
| R627 | 0.34 | 1.5 | | | 20 | 63 |
| R602 | 1.38 | 1.5 | | | 20 | 96 |
| R787 | 0.44 | 1.5 | 0.19 | | 22 | 93 |
| R789 | 0.49 | 1.5 | | 0.11 | 22 | 73 |
| R699* | 0.35 | 1.5 | | | 18 | 79 |

* Plus 6 lb per ton Lan-Cer-Amp.

Table III. Summary of Results

| Deoxidizer | Range for ~20 pct Elongation at 2400°F Wt Pct Residual | Major Type of Inclusions | Distribution of Inclusions | Structure |
|---|---|---|---|---|
| Mn | >4.25 | Chromites, Silicates | Random | Austenite |
| V | 1.45-2.60 | Vanadium chromite | Random | Austenite + Ferrite |
| Si | >1.20 | Glassy silicate | Random | Austenite |
| Al | 0.16-0.26 | Alumina | Segregated | Austenite |
| Ce | 0.05-0.10 | Glassy silicate | Random | Austenite |
| | >0.10 | Oxide | Segregated | Austenite |
| Ti | 0.05-0.085 | Ilmenite | Random | Austenite + Ferrite |
| | 0.30-0.47 | Titanium nitride | Segregated | Austenite |
| Zr | 0.04-0.21 | Oxide | Segregated | Austenite |
| Ca | * | Glassy silicate | Random | |

* Residual Calcium not determined, but excellent ductility obtained by addition of 0.1 to 0.2 pct Ca as Ca-Si to steels with greater than 0.50 pct Si.

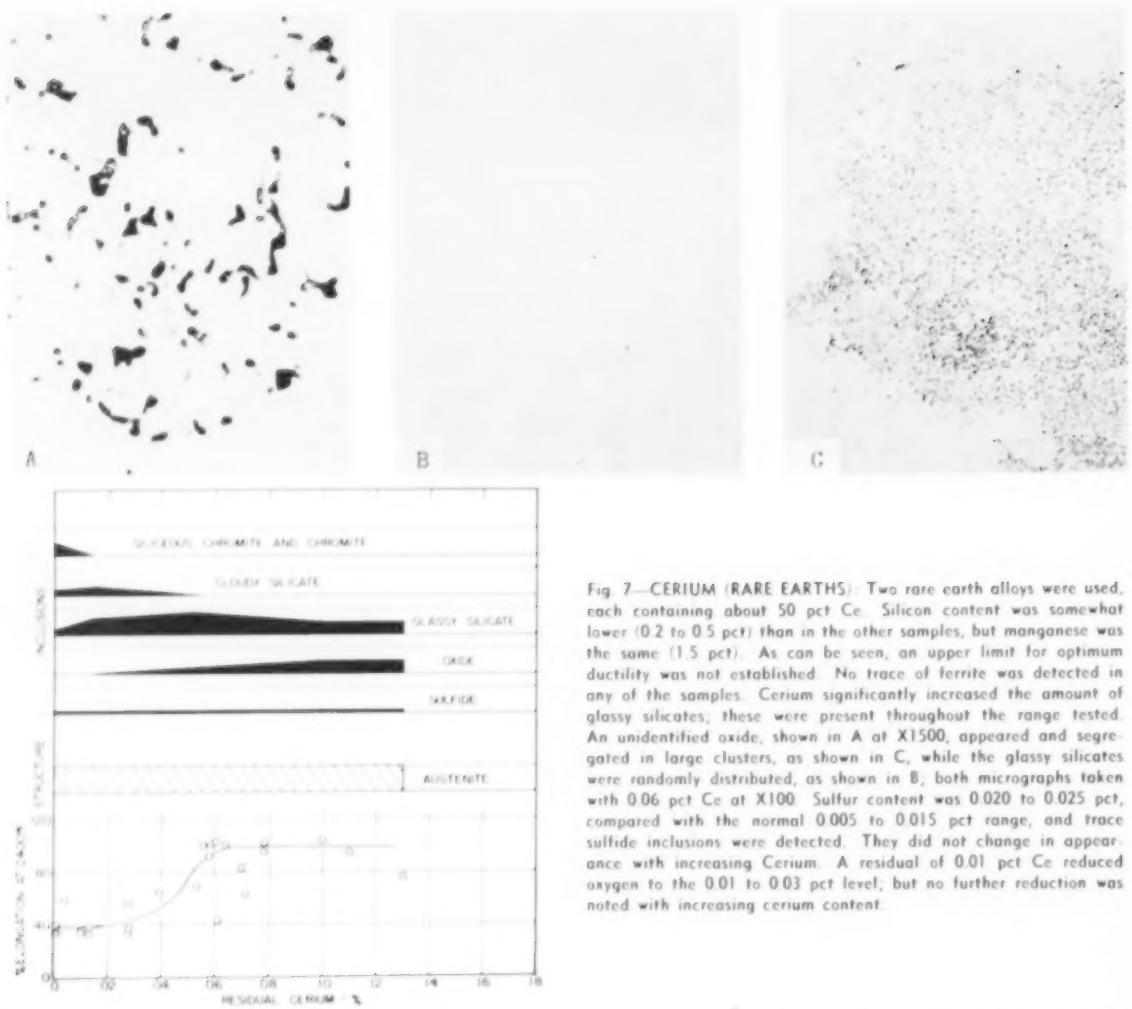



Fig. 7—CERIUM (RARE EARTHS): Two rare earth alloys were used, each containing about 50 pct Ce. Silicon content was somewhat lower (0.2 to 0.5 pct) than in the other samples, but manganese was the same (1.5 pct). As can be seen, an upper limit for optimum ductility was not established. No trace of ferrite was detected in any of the samples. Cerium significantly increased the amount of glassy silicates; these were present throughout the range tested. An unidentified oxide, shown in A at X1500, appeared and segregated in large clusters, as shown in C, while the glassy silicates were randomly distributed, as shown in B, both micrographs taken with 0.06 pct Ce at X100. Sulfur content was 0.020 to 0.025 pct, compared with the normal 0.005 to 0.015 pct range, and trace sulfide inclusions were detected. They did not change in appearance with increasing Cerium. A residual of 0.01 pct Ce reduced oxygen to the 0.01 to 0.03 pct level; but no further reduction was noted with increasing cerium content.

tion of duplex austenite-ferrite structures and indicates that too much of a deoxidizer may be as harmful as too little.

With one exception, the initial increase in hot-ductility of the steel in the hot-worked condition was accompanied by a significant change in the types of oxide inclusions present in the steel. It should not be concluded, however, that hot-ductility is controlled simply by products of deoxidation, since high ductility was observed in steels having widely different types, amounts, sizes, shapes, and distribution of inclusions. It appears more likely that the change in inclusion types reflects a fundamental change in composition or the structure of the steel which, in turn, alters hot-ductility.

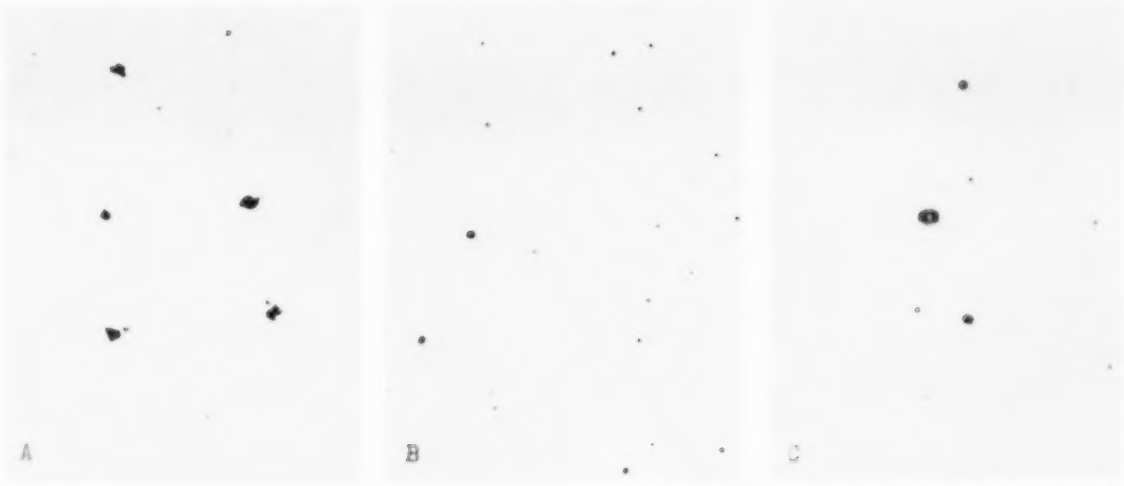
Ferrite, which formed in steels treated with vanadium, aluminum, titanium, and zirconium, initially had an anomalous effect on hot-ductility of wrought type 310 steel. The ductility of the vanadium-treated steels first increased, that of the aluminum-treated steels remained essentially constant, and that of the titanium or zirconium-treated steels decreased simultaneously with the initial appearance of ferrite in the microstructure. In all cases ductility decreased as significant amounts of ferrite formed. It appears that the manner in which this phase is formed and its distribution in steel may be more significant from the standpoint of ductility than the mere presence of ferrite in the structure. This, in general, agrees with results reported by Lula and Henke.[3]

With few exceptions, deoxidation practices used in this investigation effected significant reductions in total oxygen content and changed the nonmetallic inclusions to different types, characteristic of the specific element added. The amount required to promote this change was relatively low. Hot-ductility could not be correlated with total oxygen content.

The optimum combination of a high level of ductility at 2400°F, a fully austenitic structure, and a favorable type and distribution of nonmetallic inclusions in hot-worked steels was realized with the addition of 0.1 to 0.2 pct Ca as Ca-Si alloy. Although aluminum, titanium, zirconium, and cerium significantly improved hot-ductility, they promoted serious segregation of nonmetallic inclusions. With titanium and zirconium, segregation was associated primarily with nitride inclusions. Silicon, manganese, and vanadium produced a more random distribution of inclusions, but rather large additions of these elements were required for good hot-ductility.

## Results with Cast Steels

The effect of silicon, aluminum, titanium, and rare-earth additions on the tensile ductility of ingot



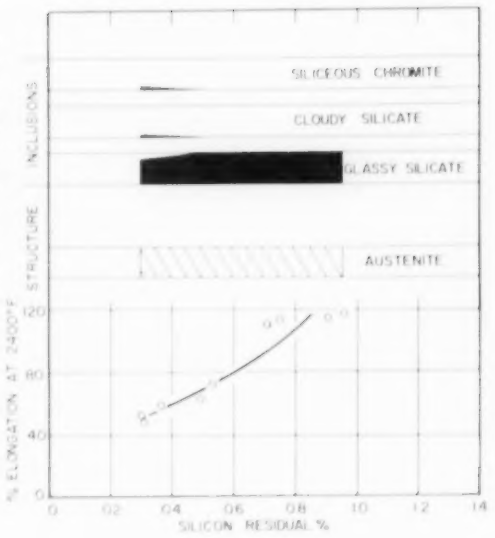




Fig. 8—CALCIUM: Due to its high vapor pressure and reactivity at relatively low temperatures, calcium must be added to steel with a carrier element to be effective as a deoxidizer. Silicon is the most commonly used carrier. Calcium was added to the steel in the form of Ca-Si alloy, a frequently used deoxidizer. It was not possible to add increased amounts of calcium and hold silicon constant at the desired 0.5 pct level due to the relatively high silicon content of this alloy. Consequently, the relative effect of calcium was determined by the addition of calcium in a narrow range to steels of widely different silicon content. The data obtained then was compared with that obtained from steels of the same silicon content but without the calcium addition. Since calcium has a very low solubility in steel, no attempt was made to analyze for the trace amounts which might be retained. Hot-ductility for a given silicon content was considerably higher in steels treated with calcium than in untreated steels. Better than 70 pct elongation was observed in steels with as little as 0.5 pct Si and an average elongation of better than 100 pct was obtained at 0.8 to 1.0 pct Si when 0.1 to 0.2 pct Ca was added as Ca-Si. Delta ferrite was not detected in any of the steels. Although calcium did not promote any new deoxidation products, glassy silicates were found at much lower silicon content when calcium was added (compare with Fig. 1). As shown above, all inclusions were randomly distributed: A—0.45 pct Si, no calcium; B—0.99 pct Si, no calcium; and C—0.49 pct Si, with 0.10 pct Ca, all at X500. Calcium-silicon additions reduced oxygen content to a much lower level than that obtained with silicon alone.

metal at 2400°F was investigated. For this study, 50-lb ingots were induction-melted and cast in 3-in. square molds. The same heat-making practice as used in preparing smaller ingots for tests on hot-worked metal was employed.

The results obtained are summarized in Table II. The additions had no effect on the hot-ductility of cast metal. All samples prepared from the ingots had relatively low ductility, which may be associated with the coarse columnar grain size. These data indicate that the results of tensile tests on wrought metal may not be directly applicable to predict the behavior of ingots during initial ingot breakdown operations.

## Significance of Results

The importance of deoxidation additions has been clearly demonstrated, and a general behavior pattern appears to exist. However, the observed relationships between hot-ductility, microstructure, or inclusions and deoxidation practice are characteristic of one grade of steel melted without slag-cover and solidified at a rapid rate. Any change from these conditions most likely would alter the relationships to some extent. It is recognized that the results of this study cannot be applied directly to commercial practice and most likely cannot be used to predict workability in ingot breakdown operations. However, the observations should have general significance and applicability to the behavior of hot-worked metal. Ultimately, the results of this study should lead to practices in the manufacture of stainless steel, which will improve resistance to hot-cracking during subsequent fabrication, and which may extend so far as to reduce the incidence of base metal cracking in the welding of restrained joints.

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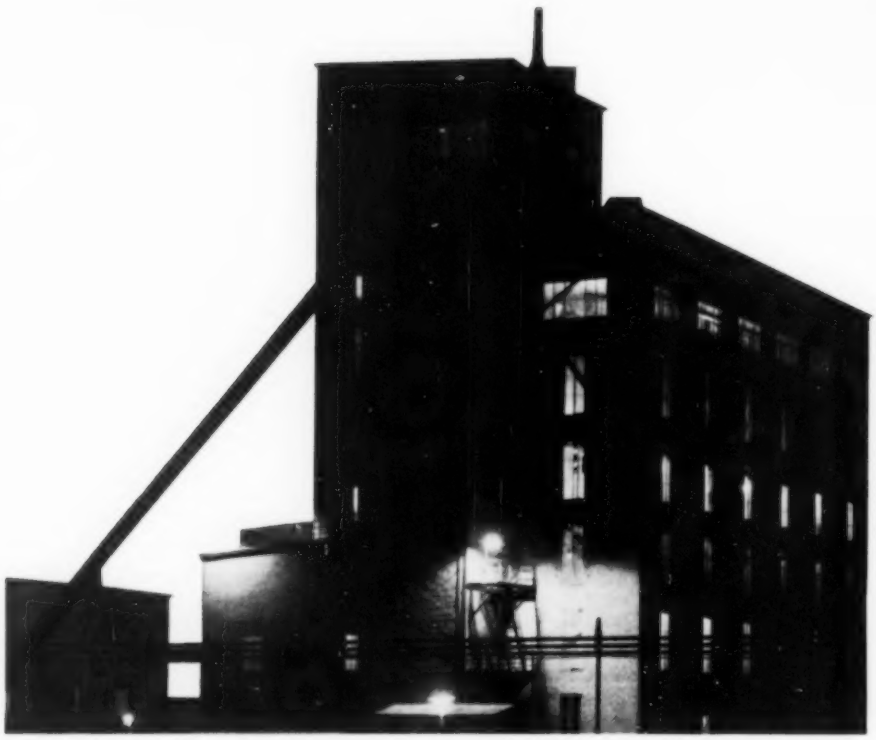
by John L. Stalhed



A typical Wiberg-Söderfors sponge iron plant in Sweden.

# Sponge Iron in Electric Arc Furnaces

*When reducing iron ores at temperatures below their melting point, the end product will be a porous mass of more or less completely reduced iron; this product is called sponge iron.*

*Sweden utilizes a greater tonnage of sponge iron than any other country at the present time. This report deals with Swedish experience and technique in the use of sponge iron in electric arc furnaces.*

SWEDEN'S steel production is, to a great extent, specialized in high grade steels, for, of the total production of ingots—about 2,500,000 tons in 1955—550,000 tons was high quality steel. Freedom from metallic impurities is very important for such special steels, because of rigid specifications. For this reason, purchased scrap is only used to a limited degree by high grade steel producers, and so-called *selected* scrap of sufficiently high purity is available only in small quantities. Circulating scrap from high grade steel constitutes only about 25 pct of the melting stock; so, a large part of the raw material demand must be covered by virgin melting stock.

Acid open hearth processes, utilizing charcoal pig iron, played an important role in the country's earlier special steel production, but at present little charcoal pig iron is produced. It has been replaced by a special coke pig iron, produced from high grade ores and low phosphorus coke. This pig iron is sometimes desulfurized, preferably with lime according to Kalling's method. An appreciable quantity of steel, therefore, can still be produced in acid open hearth furnaces, especially ball bearing steel. But the greater portion of Sweden's high grade steel production is made today in electric furnaces.

In the electric furnace steel process a charge with a relatively low carbon content is desired, for it is not considered economic to remove large quantities of carbon with oxygen in the furnace.

A low carbon charging material can be obtained in various ways. One possibility is to decarburize pig iron, for instance in a bessemer converter, but this method requires a large capital investment and can only be adopted to large-scale production. Quite often a plant needs a limited addition to its melting stock, say 50,000 tons per year. In such cases sponge iron has proved to be a good solution.

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Sponge iron fulfills the requirement of a suitable carbon content as well as high purity. Sponge iron, according to the Wiberg-Söderfors method,[1,2] is produced in Sweden at five different steel plants with a combined annual output of about 100,000 tons. In addition, about 30,000 tons is manufactured according to the Höganäs sponge iron method,[3] but information in this paper is based on experience gained in melting sponge produced according to the Wiberg-Söderfors method. However, the results are believed to be applicable to other types of sponge iron.

## Production of Wiberg-Söderfors Sponge Iron

The Wiberg-Söderfors process utilizes a shaft kiln with gases produced from coke or other fuels, while the Höganäs process uses a muffle-type kiln, in which the ore is heated in intimate contact with coke or coal.

**Ore:** For the Wiberg-Söderfors process ore requirements are as follows:

1) The lump size of the ore must be adequate, generally in a range of 25 to 80 mm;

2) The iron content should exceed 65 pct;

3) The phosphorus content must be low, since the entire amount remains in the sponge iron; and

4) The ore should possess sufficient mechanical strength during reduction.

Ore for this process can be lump ore or pellets of about 1 in. diam; the latter have proven especially suitable. Slag constituents in the pellets have a definite influence on the strength during reduction, and an addition of hydrated lime has been found to be especially favorable for maintenance of high strength. As lime normally is desired in the melting of sponge iron, it is commonly used as an addition in pelletizing in amounts from 1 to 1.5 pct.

Iron content of the ore or pellets should be as high as possible, because the gangue minerals remain in the sponge and have to be melted in the steel furnace. Generally, at least 66 pct Fe is desired if the ore is a magnetite, or 65 pct if hematite or pellets. Fig. 1 shows how the iron and slag contents of the sponge vary with the iron content of the ore.

**Costs:** The cost of production of Wiberg-Söderfors sponge iron is shown in Table I. The figures are based on several years' Swedish experience, and the cost is calculated according to American prices. The cost of production specified in Table I is obtained in units producing 20,000 to 30,000 tons per year. If larger units are built, the cost per unit output is likely to decrease.

Capital cost for a complete plant consisting of two furnace units, building, storage bins, and transport equipment, for an annual production of 60,000 tons of sponge is approximately $4 million.

**Fuel:** In the Wiberg-Söderfors process different fuels can be utilized as long as they can be brought into reaction with the circulating gas and regenerate this to CO + $H_2$. This reaction takes place in an electrically heated carburetor, filled with coke, and hydrogen is added in the form of natural gas or other light hydrocarbons or coke oven gas.

The process consumes very little fuel, in fact less than any other existing reduction process. In Sweden, propane and butane are used instead of natural gas in a coke carburetor to produce a reducing gas consisting of carbon monoxide and hydrogen in the proportion 2:1. In place of using the coke carburetor, a reducing gas could be produced from oil or natural gas according to existing methods, and in this case power consumption would be much lower.

Table I. Cost of Producing Wiberg-Söderfors Sponge Iron According to Swedish Practice and American Prices

| | | Per Ton of Sponge with 90 Pct Fe | |
|---|---|---|---|
| | | Quantity | Cost in $ |
| Pellets of 66 pct Fe | | 1.37 tons | |
| Coke | @ $15.00 per ton | 270 lb | 2.03 |
| Natural Gas | @ $0.50 per 1000 cu ft | 1550 cu ft | 0.78 |
| Power | @ $0.007 per kw-hr | 850 kw-hr | 5.95 |
| Electrodes | @ $0.25 per lb | 1.5 lb | 0.38 |
| Raw dolomite | @ $3.00 per ton | 100 lb | 0.15 |
| Miscellaneous | | | 0.25 |
| Direct labor | @ $2.75 per hr | 1 manhour | 2.75 |
| Repair and maintenance | | | 1.25 |
| Supervisory labor | | | 0.30 |
| Total, exclusive of ore, overhead, and capital costs | | | $13.84 |

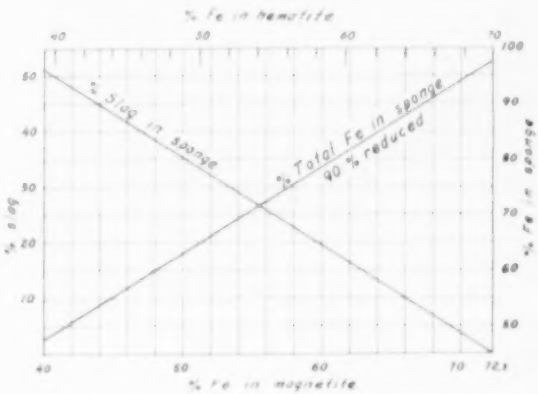


Fig. 1—To produce sponge suitable for the electric arc furnace, iron content of ore or pellets should be at least 65 pct, for all gangue is carried along into the sponge.

## Consumption of Sponge

Of the total amount of sponge produced in Sweden about one-fifth is used in acid open hearth furnaces, and a small quantity is melted in induction furnaces. No melting of sponge in acid electric arc furnaces has been accomplished, but most of the sponge is utilized in basic electric arc furnaces for high grade steel production. This electric arc furnace has proven to be the one most suitable for melting sponge iron, and various amounts of sponge can be used, up to 100 pct of the charge.

## Properties of Sponge Iron

At all existing Wiberg-Söderfors installations, pellets with about 66 pct Fe are used. A typical analysis of the resulting sponge is:

| | |
|---|---|
| Metallic Fe | 82 pct |
| Total Fe | 90 pct |
| Reduction | 91 pct |
| C | 0.9 pct |
| P | 0.010 pct |
| S | 0.010 pct |

The sponge is usually a mixture of nonbroken pellets 1 in. diam, fines, and some agglomerated pellets. The volume weight is 1.5 to 1.75 metric tons per cu meter, or 95 to 110 lb per cu ft.

## Melting of Sponge Iron

Sponge, delivered from the reduction furnace in airtight containers holding four to five tons, can be

taken either directly to the melting shop or emptied into storage bins. In the latter case, the containers must cool for 24 hr in order to eliminate the risk of ignition or reoxidation, but after being properly cooled, the sponge can be stored in open air for years without risk of oxidation. However, it has to be protected against moisture, because of water absorption and rusting.

Most of the sponge is taken directly to the melting shop in the containers, because this involves less handling. Charging the furnaces can be done according to any of the usual methods, but, if a large portion of the charge consists of sponge, top charging with a basket or charging through a hole in the roof is considered more suitable. Stora Kopparberg's experience tends to favor charging through a hole in the roof, because both radiation losses and manual labor are held to a minimum. Unfortunately, the arrangements in existing melting shops do not always permit this way of charging; however, when planning new furnaces, charging through a hole in the roof seems worth considering.

Contrary to melting scrap, the electric conditions are very stable when melting sponge; electrodes burn quietly, and the load is constant. Full power can be applied from the beginning. The electrodes are submerged in the sponge during the main part of the melting period. Owing to this, the heat losses are decreased, and the roof is protected. The electrical conductivity of sponge with 90 pct Fe is good, in spite of its relatively low volume weight. No difficulties have been encountered in obtaining contact with electrodes. The heat conductivity of sponge iron is low, because of its porous structure, but this is no disadvantage in an arc furnace.

During the melting, a gas, consisting of CO and $CO_2$, is evolved from the sponge, because of the reaction between FeO and precipitated C in the sponge. This gas protects the charge from oxidation, and it also permits melting to take place in a nitrogen-free atmosphere.

If the gangue minerals are highly acid, extra lime is added together with the sponge. Normally, a $CaO:SiO_2$ ratio of 1.2 to 1.7 is best in the gangue slag. The FeO content of this slag is usually 7 pct.

Some examples of the procedure and results at two different steel works that regularly use sponge iron are given below:

**Example I**

Furnace capacity: 13 tons
Shell size: 15x11 ft, oval
Electrodes: 4 top electrodes 9 in., bottom electrode
Transformer: 4000 kva, Scott connected

Various kinds of high grade carbon steels and low alloy steels, mostly tool steels, are produced. The charge consists of various quantities of sponge, often 70 pct, plus home scrap and some pig iron; no purchased scrap is used. Heats with 100 pct sponge also have been made with no difficulty.

The sponge is transported in steel containers, each taking 3.6 tons of sponge. Scrap and pig iron are charged first through the door with a small charging machine. After that, one container of sponge is charged through a 1-ft diam hole in the roof. It takes about 20 sec for the sponge to flow into the furnace. The power is switched on, and after about 1 hr, when the charge is partially melted, another container of sponge is charged. At the same time, some of the gangue slag is tapped through the door. When the second quantity of sponge is melted—after another 45 min—a third sponge container is charged in the same way. After all of the charge is melted—a total of 3½ hr—slag is tapped and raked. Some of the gangue slag is kept in the furnace and enters into the final slag.

A melt-in carbon content of 0.7 pct is desired. Generally no refining is done, but, in spite of this, very high quality steel is obtained. The heat is finished under a reducing slag.

Analysis of the sponge charged is as follows: metallic Fe 77.5 pct, total Fe 86 pct, 90 pct reduction, 0.9 pct C, 0.015 pct S, 0.010 pct P, and 7.5 pct gangue.

The $CaO:SiO_2$ ratio in the gangue is about one, and no extra lime is added with the sponge. No damage of the lining from the sponge has been observed, even when sponge is charged directly on the bottom. Table II gives the production data for Example I.

**Example II**

Furnace capacity: 33 tons (Demag)
Shell diam: 16 ft
Transformer: 9000 kva, max 280 v
Three Electrodes
Top charging
Product: Killed steel of 0.10 pct C for tubes
Two slags

Sponge iron is charged in various quantities, together with pig iron, home scrap, and purchased scrap. For steels of high purity, up to 70 pct of sponge is charged. Analysis of sponge charged is as follows: metallic Fe 77.3 pct, total Fe 90 pct, 86 pct reduction, 0.8 pct C, 0.002 pct P, 0.007 pct S, and 5.6 pct gangue.

The gangue is rather acid with the $CaO:SiO_2$ ratio 1:3. Scrap and pig iron are charged first, and sufficient lime is added together with the sponge to give a $CaO:SiO_2$ ratio of 1.7 in the slag. Sponge is never charged directly on the bottom, because the acid

Table II. Production Data of Example I, Melting in a 13 Ton Arc Furnace

| No. of Heats | Pct Sponge in Charge | Power Consumption, Kw-Hr per Ton | Total Time, Hr | Production, Tph |
|---|---|---|---|---|
| 26 | 23.9 | 719 | 6.57 | 2.01 |
| 58 | 47.5 | 813 | 6.72 | 1.90 |
| 61 | 67.6 | 851 | 6.97 | 1.94 |

Avg yield of ingots 97.1 pct
Consumption of lime for final slag, 50 lb per ton of steel
Consumption of electrodes, 12.4 lb per ton of steel

Table III. Production Data for Example II, Melting in a 33 Ton Arc Furnace

| | | | | |
|---|---|---|---|---|
| Charge: | Pig iron | 11* | 11* | 21* |
| | Home scrap | 28 | 41 | 25 |
| | Purchased scrap | 61 | 46 | 0 |
| | Sponge iron | 0 | 12 | 54 |
| Total Fe charged, tons | | 36.70 | 36.34 | 36.42 |
| Ingots produced, tons | | 34.07 | 34.00 | 34.78 |
| Yield, Pct | | 92.9 | 93.6 | 95.5 |
| Time from previous tapping to melt, hr | | 2.83 | 3.08 | 3.63 |
| Refining period, hr | | 2.25 | 2.15 | 2.22 |
| Total time, hr | | 5.08 | 5.23 | 5.85 |
| Production, tph | | 6.70 | 6.51 | 5.95 |
| Lime, lb per ton | | 78 | 78 | 127 |
| Power consumption: | | | | |
| For melting, kw-hr per ton | | 676 | 611 | 622 |
| Total | | 695 | 631 | 745 |

* Each group is an avg of 20 heats.

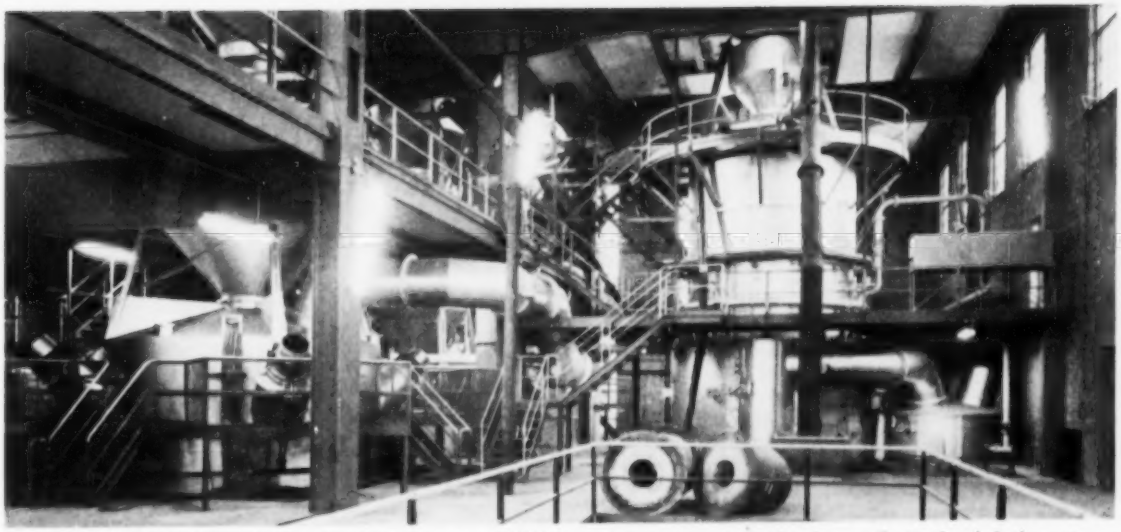

Fig. 2—At this Wiberg-Soderfors plant in Sweden some 30,000 tons of sponge iron is produced annually in the shaft furnace shown at right. The coke carburetor, at left, furnishes the reducing gas.

gangue in this sponge damages the lining. Experience has shown that the sponge, when charged, should not be allowed to pile up on the furnace walls, as there is a tendency for it to stick to the sidewalls. If this occurs, clumps of oxidized sponge may drop into the bath, causing very strong evolution of gas. Table III gives the production date for Example II.

**Examples I and II:** The yield in both of the foregoing examples is calculated from the weights of pig iron plus scrap plus total Fe content in the sponge.

In Example I, the melting time for sponge heats and scrap heats is about the same. This is due primarily to shorter charging time and better heat economy through the utilization of *top hole* charging, and, to a lesser extent, to the utilization of a well reduced sponge.

In Example II, the melting time is longer for sponge heats than for scrap heats. However, this is often compensated for by shorter refining time.

Due to the content of gangue and non-reduced FeO in the sponge, power consumption for melting sponge must necessarily be higher than for melting scrap. Assuming 85 pct efficiency, the extra power required for melting sponge can be calculated to be 0.312 kw-hr per lb of slag plus extra lime, and 0.433 kw-hr per lb of Fe combined with oxygen as FeO. For a good quality sponge iron, this extra power required is small, and for a sponge with 90 pct total Fe, 91 pct reduction and 7.5 pct slag, it is 130 kw-hr per ton of Fe.

These figures illustrate the importance of a low gangue content and a high degree of reduction. Therefore, an ore with the lowest possible gangue content should be used as a raw material for making sponge iron. Moreover, experience has shown that the lower the gangue content in the ore, the higher will be the degree of reduction obtained in the sponge.

## Summary

The advantages of introducing a pure raw material, such as sponge iron, in the steel process can be summarized as follows:

1) Higher yield in the steel furnace;

2) Higher yield in the rolling mill because of better rolling properties;

3) Fewer rejects; and

4) Plant circulating scrap lower in impurities, especially Cr, Cu, and Sn, which tend to accumulate in the scrap.

The advantages of using sponge have been found to be so important that they fully justify its use, even when scrap is cheaper than sponge, as is now the case in Sweden. This also compensates for a somewhat higher melting cost in the electric furnace. However, if the sponge is well reduced, say 92 pct reduction, and has less than 2 pct $SiO_2$, then the cost of melting should not be higher than for scrap. A technically sound procedure is to use a combination of hot metal and sponge iron. In this case a lower degree of reduction of the sponge can be tolerated and is even desired.

So far, sponge iron is used in Sweden only for high grade steel. However, in spite of the fact that it is produced only in small quantities, the production cost is not higher than for pig iron. In addition, sponge iron offers the possibility of permitting coke to be replaced by natural gas or oil as a reduction medium. The production of sponge iron in large quantities for commercial steelmaking, therefore, seems worth considering.

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# A Program for Fundamental Research In Hard Metals

by Pol Duwez

HARD metals constitute a rather unusual class of solids for the physical metallurgist interested in the most fundamental properties of the solid state.

**Hard metals are essentially intermetallic compounds.**

But they are unusual ones, because they contain two kinds of atoms which are extremely different in their electronic configuration. One atom is what may be called a true metal having the most typical metallic bond, and the other is a nonmetallic element. The metallic element in hard metals is generally an element of the transition groups of the periodic table, and the nonmetallic element may be carbon, nitrogen, or boron. From the fundamental point of view, the term hard metal could also be applied to silicides, and if so, germanium and tin should also be considered. In addition, the oxides of the transition metals belong to the same general class, although the practical metallurgist would probably not agree, since most of these oxides are not hard enough to be compared with the carbides. We can, therefore, define the general field of hard metals as compounds between true metals—mostly those of the transition series—and atoms which generally behave like interstitials when they are soluble in the metals.

Because of the large difference between the electronic configurations of the two constituent atoms, atomic bonds involved in hard metals are necessarily a complex and controversial question. What are now called the theories of atomic bonding in hard metals are not much more than phenomenological explanations of some of the properties of these materials. The basic difficulties involved in this problem are due to the existence of metal to metal, metal to nonmetal, and nonmetal to nonmetal bonds.

The problem of bonding in refractory hard metals is obviously a difficult one, and the reason may be that all the possible transitions between different bond types from ionic to covalent to metallic are involved in these structures. To sum up, it is not unfair to say that,

**Our theoretical explanation of the atomic bonding in hard metals is still in an embryonic form . . . What can be done to improve this situation?**

It would be difficult to convince the theoretical physicists that hard metals should receive their immediate attention. They probably find the problem too complex and postpone its solution until other simpler problems have been worked out. In the meantime, should the metallurgist relax and wait?

No, metallurgists should not cease all activities directed toward a better understanding of our solid state problems. We should design suitable experiments which ultimately will be of great value in deciding how close the physicist's calculations can come to a perfect interpretation of nature. Good measurements of physical properties of solids are always useful, and by detecting some correlations between these measurements, new concepts may be formulated which sooner or later will be helpful to the theoretical physicist. Progress in the understanding of alloys during the past 25 years is the result of such an empirical approach. It is after having observed analogies between some simple systems involving copper, silver, and gold as the solvent, that Hume-Rothery formulated his basic concepts and his empirical laws of alloying which are now classical.

**The most basic measurements should be made on single crystals.**

Properties of single crystals are physical constants. Measurements of the elastic constant of crystals, for example, are of the utmost theoretical importance in connection with the strength of the atomic bond. Such measurements have been made on pure metals and also on ionic crystals such as NaCl. These experiments are not easy to perform, and for hard metals the experimental difficulties are greater by at least one order of magnitude. Hard metal single crystals, large enough to be subjected to physical property measurements, cannot be produced with the presently known techniques. But it should never be said that no solution exists for this problem. Either new techniques should be developed by which measurements can be made on microscopic crystals—of about 1 mm or less—or the presently known methods of growing single crystals should be improved so that much higher temperatures can be reached.

**But instead of single crystals, sintered material must be used at present.**

In the case of sintered material, the most bothersome factor involved in the measurements is porosity of the specimen. Because of voids in the solid, a large portion of the data reported in the literature on elastic modulus, electrical and thermal conductivities, etc., are, if not completely useless, at least very difficult to interpret. But though such measurements are useless in the theoretical sense, they may be very useful in application.

We should, therefore, try to achieve 100 pct density, and the best way to approach this goal seems to be hot pressing. One way of obtaining useful data with materials which are not 100 pct dense is to measure the variation of a physical property with another factor such as temperature. If, for example, the electrical resistance of a specimen (not the resistivity which is density dependent, but the resistance) is measured over a range of temperature, the slope of the curve so obtained may have

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great theoretical significance. It is important not to restrict the temperature range over which variations of physical properties are measured. There are many difficulties involved in doing this, but these should be minimized in the near future, when the facilities of modern metallurgical laboratories include cyrogenics as well as high temperature sections.

### Some special features of hard metals should be considered in designing useful experiments.

It is well known that hard metals may exist over a wide range of compositions with either a deficiency or an excess of one of the two components. In the case of titanium carbide, for example, as much as one carbon out of two may be absent from the lattice. These vacancies in the crystal structure have a pronounced effect on some physical properties, and quite a few interesting studies have been made on this subject. The same situation is found in oxides (for example, TiO), and here it is generally assumed that the presence of vacancies changes the valence of the ions in the neighborhood of the vacancy. What is happening in a carbide or a nitride is not clear, but since oxygen and carbon can substitute for each other in the NaCl structure, perhaps a systematic study of the solid solution TiO-TiC with and without vacancies would throw some light on the subject. This study would, in fact, cover the field of the Ti-O-C ternary diagram in which the cubic NaCl phase exists. In connection with the study of oxygen-deficient oxides, paramagnetic resonance spectroscopy perhaps could furnish some information on the exact nature of the change in ionization with the removal of oxygen. Paramagnetic resonance is a new tool, and this is what is needed to uncover new facts.

When oxygen and carbon replace each other in a binary system, such as TiO-TiC, the bond changes progressively from a typical ionic bond to what may be called a carbide bond. Such systems should be most interesting to study, but many similar systems can be found between carbides, nitrides, hydrides, borides, oxides, and perhaps silicides. In addition, metals also can replace each other in a given hard metal, and systematic investigations of the properties of properly selected systems may lead to interesting observations in comparing the behavior of metals of the same group of the periodic table in a given crystal structure.

In many of the high temperature applications of nonmetallic materials, stability is a very important requirement. Progressive deterioration of the material may be produced by reaction with a gas, a liquid, or another solid in close contact. In most cases, solid state diffusion is the most important controlling factor in this problem, and, hence, diffusion in hard metals is of paramount importance. Metallurgists can claim that they now have a good understanding of the mechanism of diffusion in metals and alloys in which either substitutional or interstitial atoms are involved. Also, much progress has been made in explaining quantitatively the role of solid state diffusion in gas-metal reactions.

### Testing of single crystal specimens is foreseen.

Although there is no method for making diffusion experiments on a crystal which is smaller than 1 cu mm, such experiments are not impossible. In chemistry, the size of an analytical sample has been pushed down beyond the mg scale. In metallurgy, techniques have been devised for the determination of the mechanical properties of whiskers which are in the micron range. There are physical methods by which an area as small as 1 $\mu$ of a metallic surface can be quantitatively analyzed—the microelectronic probe, where a beam of electrons can probe an area of 1 $\mu$. Micro X-ray beams for X-ray diffraction studies and radioactive tracers seem to lend themselves very well to microscopic samples. Paramagnetic resonance spectroscopy, mentioned earlier, is also adaptable to small specimens. This problem of microtesting will be solved by the young generation of physical metallurgists who, without any preconceived ideas, will attack the problem of measuring, for example, the modulus of elasticity of a single crystal of titanium carbide not bigger than a pinhead.

### Cemented structures present even greater difficulties.

This discussion has been limited to pure hard metals or their mixtures. When a metallic binder is present, the problem becomes considerably more difficult to analyze theoretically. The structure of a cemented tungsten carbide, or of a chromium-alumina cermet, consists essentially of two distinct phases, with a fine layer of some more complex material between the two phases. To predict the physical properties of such a solid material, knowing those of the two constituents is, at the present time, impossible. Perhaps the most difficult factor to introduce in such an analysis is what might be called the geometrical factor, which would take into account the size and the shape of the two phases. There is a definite need for systematic experiments using simple systems which may not be of any practical interest, but in which the geometry of the two phases could be accurately controlled and accurately described over a wide range of compositions. Providing also that these cermets can be made with 100 pct density, measurements of electrical resistivity, thermal conductivity, thermal expansion, and modulus of elasticity should be most interesting. Starting from an idealized mechanical model, properties of two-phase alloys can be computed knowing the properties of the constituents. Experimental results are needed to decide whether or not approximate computations, based on a simplified mechanical model of a two-phase alloy, have any practical value.

### Fundamental research will help solve practical problems.

The author's emphasis is on the fundamental approach to the study of hard metals. But in an industrial research laboratory, the solution of immediate problems is very often of primary importance. In trying to develop a hard metal or a cermet for a specific high temperature application, such as a fuel element, or a gas turbine blade, no one expects to be able to design the material by solving complicated equations, even if the most elaborate computing machines are available. The conditions to which the material will be subjected must be analyzed, the constituents of the cermet chosen on the basis of their physical properties, and good judgment used. Most of the physical properties which are needed in guiding the proper choice are still lacking.

Therefore, the author must emphasize that a systematic study of physical properties of hard metals will help the practical metallurgist as well as the theoretical physicist.

# The Steel Industry and International Flame Research

From an Article by M. Riviere

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the United Nations' Economic Commission for Europe in Geneva, Switzerland, 1956.

**Steel industries of several western European countries—Netherlands, France, the United Kingdom, Belgium, and Sweden—as well as the United States and the High Authority of the European Coal and Steel Community, have for some years been taking part in a general research project on the properties of flames used in industry, under the aegis of the International Committee on Flame Research, at an experimental research station at Ijmuiden, Netherlands. The results of the work are published as research proceeds, but it is only in the last two years, and especially in 1955, that the first effects of this work on the productivity of the steel industry have begun to appear.**

INTERNATIONAL Flame Research has not been conducted with any particular application in view, since many industries, including glass, cement, oil, coal, gas, and furnace manufacturers, have participated. It was, however, the low efficiency of the open hearth furnace, in which only about 20 pct of the heat is absorbed by the charge, that was the initial stimulus for the project. The first application of this research, thus, has been the open hearth furnace of the steel industry.

### Heat Transfer in Open Hearth Furnaces

Research workers formulated a theory of heat transfer in open-hearth furnaces, so that it has now become possible to develop a general theory,[1] taking as basic data results directly derived from work carried out by the International Committee on Flame Research. This theory provides engineers with extremely useful information on the influence of certain parameters which determine the functioning of open-hearth furnaces, but it is based on too many unproved hypotheses to be regarded as finally established. It is, thus, necessary to continue tests on open-hearth furnaces in order to establish, in each case, the value of certain modifications designed to increase their productivity.

Increasing the carburization of the coke-oven gas by the use of fuel-oil or tar greatly increases the radiation of the flame and, simultaneously, the production of a given furnace. International Flame Research has confirmed this fact and also has shown that there is no need to increase the carburization beyond a certain limit. For an open-hearth furnace with a 100 ton capacity, the limit may be taken as about 50 pct of the total calories delivered to the furnace; beyond this figure, the radiation produced by the flame does not increase any further when the percentage of fuel-oil calories is raised. It must be pointed out that this carburization limit is higher for a furnace of smaller capacity and, conversely, diminishes for larger furnaces; in a 200-ton furnace, for instance, little is likely to be gained by carburizing beyond 35 pct. This fact has been usefully applied in a number of European steel works during the last few years.

Although studies on the influence of the fuel used in the heating of open-hearth furnaces may be valuable, it is obvious that the choice of fuel is often dictated by local economic conditions. In most cases it is a matter of getting the best out of certain fuels which have to be used. Here again there is much to learn from the Ijmuiden studies.

### Rate of Combustion Studied

These studies have shown that the rate of combustion of the fuel and, to a certain extent, the rate of energy release from flames burning rich fuel, depends essentially on the rate of flow of the air for combustion. Since the rate at which the air is drawn in by the flame depends directly on the flow per second which the jet can deliver, it is important to make this rate of flow as high as possible. In the case of coke-oven gas, the rate of flow depends directly on the pressure at which the gas is delivered to the burner; hence, the advantage of having coke-oven gas under pressure—1 atm gage—as some steel works already do.

In liquid fuel flames, the rate of flow depends on the pressure of the atomizing agent—air or steam—at the burner; the importance of obtaining high rates of flow, thus, made it necessary to carry out a study of injection burners. This study, which was made in Britain, has resulted in the production of a burner which, as far as large flow jets are concerned, has a higher efficiency than most of the burners used in steel works up to the present. The use of this burner has become general in British steel works in the last two years. Moreover, tests carried out in a French steel works in 1955 made it possible to increase the output of an open-hearth furnace by 8.5 pct, by raising the pressure of the atomizing air at the burner from 5.5 kg per sq cm to 8.5 kg per sq cm. Similar experiments have been made in Britain and in the Netherlands.

The use of liquid fuels in open-hearth furnaces raises a controversial point: should the fuel be atomized by air or by steam? Here again, it is obvious that economic conditions alone may impose an answer. Nevertheless, in the last few years it has been possible to obtain more precise data concerning the respective advantages of these two atomizing agents. As regards flame properties, the Ijmuiden tests have shown that, as compared with

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atomization by air, the use of steam decreases the radiation of the first part of the flame, but leaves the radiation at the end unchanged.

Hence, it appears that, as regards heat transfer alone, air is better than steam. But in the open-hearth furnace another factor intervenes: the extent of the flame's reducing properties with respect to the charge. Thus, in tests carried out in a French steelworks, an increase of about 30 pct in radiation from the flame was observed when changing from steam to air atomization. But it was also shown that the amount of pig iron charged could be reduced by 40 pct as a result of the lesser degree of oxidizing obtained with air.

The Ijmuiden tests have shown that an increase of 30° to 40°C in flame temperature is obtained from a 100°C increase in the temperature of the preheated air, without any apparent change in the emission factor. Since the energy radiated by the flame increases as the fourth power of the temperature, it is natural that the tests recently carried out on an open-hearth furnace should have shown increased transfer of heat from the flame to the charge, resulting in reduced time for the operation and reduced consumption of fuel per ton of steel produced.

### Measurements to Discover Precise Working of Furnace

Although the results described above may not all be entirely original, there is one aspect of the work done on open-hearth furnaces in the last two years which is a great innovation: the large number of measurements made on these furnaces. We are not referring here to measurements for regulating fluids, but to measurements made with a view to discovering the precise working of the open-hearth furnace. It was not until 1955 that an article could be written on the *Anatomy of the Open-Hearth Furnace*[2]—an anatomy which was revealed by the recent discovery of the necessary *scalpels*. The important part played by the International Committee on Flame Research, both in the development of these instruments and in the popularization of their use should also be stressed. Special mention must be made of the now frequent use in steel works of suction pyrometers for measuring the primary air temperature (giving not only the average temperature, but also the temperature distribution in the air uptakes); in some cases it has been possible to measure the temperature of the smoke leaving the furnace. Much progress also has been made in continuous or semi-continuous sampling of smoke with a view to combustion control. Finally, there are the numerous measurements of the radiation produced by the flame in an open-hearth furnace under different working conditions; these measurements have been made in British, French, and Netherlands steel works, using pyrometers based on those developed at Ijmuiden.

The use of models simulating the production conditions of a furnace has considerably increased in the last few years. This system provides a quick means of observing many phenomena relating to combustion or to premature wear in certain areas of the furnace, thus showing what changes are needed to obtain the desired results. A recent application of this method of research in Britain has made it possible to link the wear of refractories at certain points directly with the quantity of iron oxides deposited there.

### Formulating Principles to Improve Productivity

This whole group of new measuring techniques and the results obtained by them are contributing to the formulation of common principles for improving the productivity of open-hearth furnaces. It is, of course, evident that these principles must be differently applied, according to the nature of the metallurgical problems to be solved and the economic factors in each country and even in each steel works. The fact remains, however, that the lessons learned through the studies of the International Committee, and the numerous contacts between specialists on these questions which it makes possible, lead to better direction of the research to be conducted in each case.

*Further details on International Flame Research are to be found in ASME paper no. 56—A-111.*

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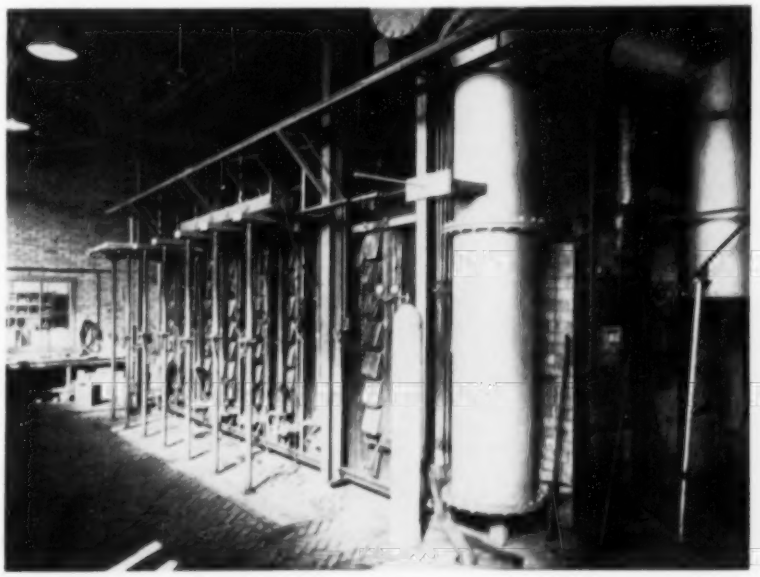
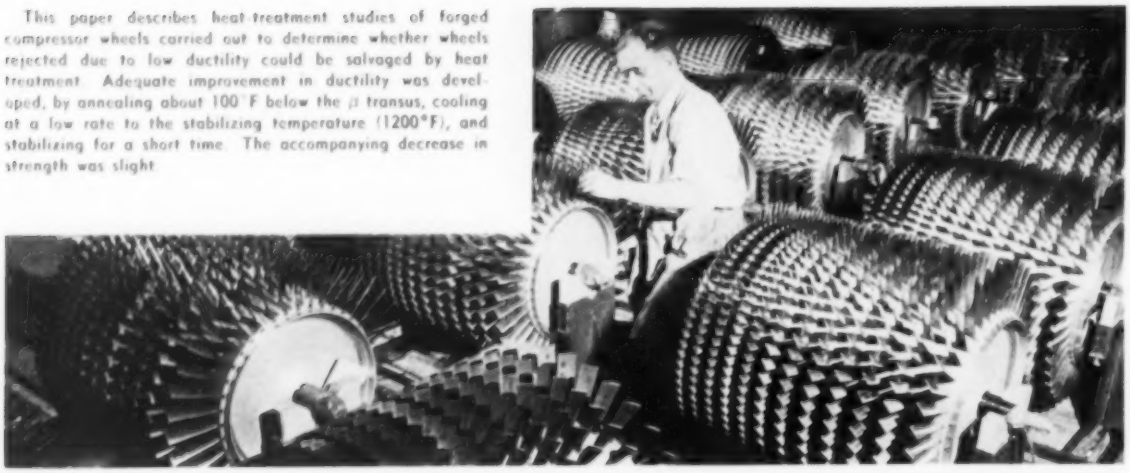


This experimental open hearth furnace at the Ijmuiden, Netherlands laboratory of International Flame Research Foundation is equipped for gas and oil studies. The laboratory also has a pulverized coal furnace. A series of ports is provided in one wall of each furnace through which radiometers can be moved up or down for a vertical traverse of the flame.

This paper describes heat-treatment studies of forged compressor wheels carried out to determine whether wheels rejected due to low ductility could be salvaged by heat treatment. Adequate improvement in ductility was developed, by annealing about 100°F below the $\beta$ transus, cooling at a low rate to the stabilizing temperature (1200°F), and stabilizing for a short time. The accompanying decrease in strength was slight.



# Heat Treatment Restores Ductility To Forged Titanium Alloys

by

D. N. Williams and R. I. Jaffee

FORGED titanium-alloy compressor wheels are used in the twelve-stage compressor unit of the J73 aircraft jet engine manufactured by the Aircraft Gas Turbine Div. of the General Electric Co. The compressor wheels are flanged disks upon which the compressor blades are mounted. The first and twelfth stages are made of 4340 steel alloy forgings, while the remaining ten compressor wheels are made of titanium-alloy forgings. Wheels machined from Ti-140A alloy (Ti-2 pct Mo-2 pct Cr-2 pct Fe) are used in the second through fifth stages, and RC-130B alloy wheels (Ti-4 pct Al-4 pct Mn) are used in the remaining stages and occasionally as replacements for Ti-140A wheels in earlier stages. Each titanium-alloy compressor wheel weighs approximately 100 lb as forged. Successive stages require large diameter wheels, so that the forging reduction increases with the intended stage.

Before acceptance, the forged and stabilized compressor wheels are required to pass a minimum tensile specification, as measured with four test specimens cut from an integrally forged test ring on the compressor-wheel rim. The tensile requirements are: ultimate tensile strength, 130,000 psi min; 0.2 pct offset yield strength, 120,000 psi min; and elongation in 1 in., 15 pct min. Rejection of the compressor-wheel forgings on the basis of insufficient ductility was excessive. Higher stage Ti-140A wheels were particularly troublesome, and the rejection rate of fifth-stage Ti-140A compressor wheels exceeding 80 pct.

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### High rejection rate made reclamation of low-ductility compressor wheels desirable.

Using material from rejected forgings, a number of heat treatments aimed at increasing ductility above the specified minimum were investigated. Although reclamation of Ti-140A forgings was of principal interest, parallel heat-treatment studies were carried out on rejected material from a RC-130B forging, to determine if the response to heat treatment of the two alloys was similar. Thermal stability of both alloys after heat treatment and the effect

Fig. 1—Results of heat treatment of alloy Ti-140A, showing $\alpha$ particles in a $\beta$ matrix. Treatment includes equilibration anneal, furnace cool, and stabilization anneal in vacuum. Etched in 1½ pct HF, 3½ pct $HNO_3$, $H_2O$ solution. X500

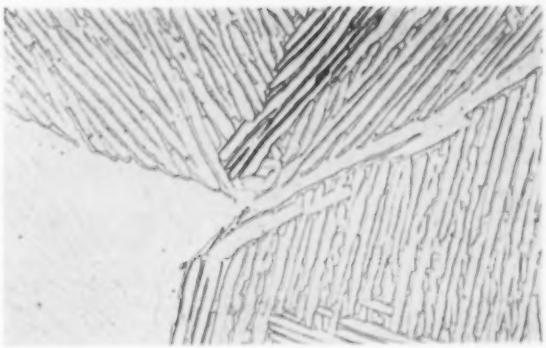

Fig. 2—Results of heat treatment of alloy RC-130B, showing large interwoven $\alpha$ needles in a matrix of $\beta$. Specimen was given an equilibration anneal, furnace cool, and stabilization anneal in vacuum. Etched in 1½ pct HF, 3½ pct $HNO_3$, $H_2O$ solution. X500

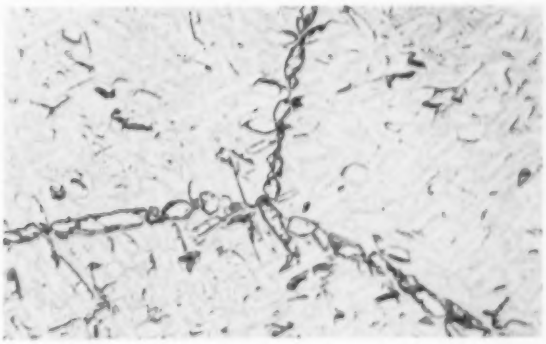

Fig. 3—Unidentified phase at the prior $\beta$ grain boundaries of air-treated Ti-140A alloy, after exposure for 200 hr at 800°F. Etched in 1½ pct HF, 3½ pct $HNO_3$, $H_2O$ solution. X500

of hydrogen content on various properties of Ti-140A were also investigated.

**Materials for Studies: Two rejected compressor wheels, one of Ti-140A alloy and one of RC-130B alloy.**

The chemical compositions of these wheels are given in Table I. The RC-130B wheel was quite close to the nominal 4 pct Al-4 pct Mn composition, and possessed a low hydrogen content. The Ti-140A wheel, although close to the nominal 2 pct Fe-2 pct Mo-2 pct Cr composition, had a high hydrogen content. The tensile properties of the forged and stabilized wheels, as measured using tangential test specimens cut from the wheel rim, are also shown in Table I.

The compressor wheels were forged from 7-in. diam billet slices, using one blocking and one finishing operation, air cooled from the forging temperature, and subsequently stabilized by annealing for 24 hr at 1200°F. Metallographic examination indicated that the Ti-140A wheel was forged at a temperature high in the $\alpha$-$\beta$ field, while the RC-130B wheel was forged in the all-$\beta$ field. The acicular $\alpha$ configuration resulting from air cooling from the

Table I. Analyses and Tensile Properties of Forged Titanium Alloy Compressor Wheels

| Material | Chemical Composition, Wt Pct | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | C | N | O | H | Cr | Mo | Fe | Al | Mn |
| Ti-140A compressor wheel | 0.06 | 0.029 | 0.25 | 0.029 | 2.04 | 1.79 | 2.24 | — | — |
| RC-130B compressor wheel | 0.10 | 0.03 | 0.16 | 0.005 | — | — | — | 4.0 | 3.9 |

| Material | Ultimate Tensile Strength, Psi | 0.2 Pct Offset Yield Strength, Psi | Elongation, Pct in 4 D | Reduction in Area, Pct |
|---|---|---|---|---|
| Ti-140A compressor wheel | 140,800 | 133,100 | 12.0 | 12.7 |
| RC-130B compressor wheel | 146,200 | 135,400 | 7.8 | 10.3 |

all-$\beta$ field was apparently the chief cause of the low ductility of the RC-130B wheel.

The rim section of the forged wheels was cut to obtain tangential bars measuring 3x½x½ in. for the heat-treatment studies. Additional bars were cut from the web section of the Ti-140A wheel for use in thermal stability tests. Material from the rim was stronger and more ductile than material from the web, possibly due to differences in the forging reduction in the two regions of the wheel.

### Beta transus first determined.

Before a heat-treatment program could be developed, it was necessary to determine the $\beta$ transus of material from the wheels. Sections cut from the rim section of the wheels were hot rolled to sheet in the $\alpha$-$\beta$ field for this purpose. Ti-140A material was rolled to 0.10 in. at 1450°F, using reductions of 20 pct per pass. RC-130B material was rolled to 0.04 in. at 1500°F, using reductions of about 15 pct per pass. The sheet was cut into small samples and solution annealed in air for ½ to 1 hr at 25°F intervals between 1450° and 1675°F for Ti-140A, and 1600° and 1750°F for RC-130B. At the end of the solution anneal, the samples were rapidly quenched in water and examined metallographically for evidence of untransformed (equilibrium) $\alpha$.

The temperatures of the $\beta$ transus of the wheels, as determined by these tests, were 1650°F for the Ti-140A wheel and 1690°F for the RC-130B wheel.

### Heat treatment gives information on variables.

A heat-treatment program was designed to give information on a number of variables. A complete description of the heat treatments, along with tensile results and hydrogen content, is shown in Table II.

**Table II. Heat Treatments, Avg Tensile Properties, and Hydrogen Content of Forged Titanium Wheels**

| Heat Treatment | | | | | | | | | | Tensile Properties | | | Hydrogen Content |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Annealing | | | Stabilizing | | | | | | | |
| Type | Atmosphere | Heating Rate[1] | Time, Hr | Temp, °F | Intermediate Cooling Rate[2] | Time, Hr | Temp, °F | Final Cooling Rate[3] | | 0.2% Yield Strength, psi | Ultimate Strength, psi | Elongation, pct in 2 in. | ppm |
| **Ti-140A Wheel** | | | | | | | | | | | | | |
| As received | — | — | — | — | — | — | — | — | | 135,000 | 145,000 | 15 | 50 |
| Isothermal equilibration anneal | Air | Rapid | 1 | 1300 | — | — | — | Water quench | | 125,000 | 135,000 | 15 | 50 |
| | Air | Rapid | 4 | 1300 | — | — | — | Water quench | | 125,000 | 135,000 | 15 | 50 |
| | Air | Rapid | 24 | 1300 | — | — | — | Water quench | | 125,000 | 135,000 | 15 | 50 |
| | Air | Rapid | 1 | 1400 | — | — | — | Water quench | | 125,000 | 135,000 | 15 | 50 |
| | Air | Rapid | 4 | 1400 | — | — | — | Water quench | | 125,000 | 135,000 | 15 | 50 |
| | Air | Rapid | 1 | 1500 | — | — | — | Water quench | | 125,000 | 135,000 | 15 | 50 |
| | Air | Rapid | 4 | 1500 | — | — | — | Water quench | | 125,000 | 135,000 | 15 | 50 |
| Isothermal equilibration anneal followed by furnace cooling to stabilizing temperature | Air | Rapid | 1 | 1300 | Furnace cool | 1 | 1200 | Air cool | | 125,000 | 135,000 | 15 | 50 |
| | Air | Rapid | 4 | 1300 | Furnace cool | 1 | 1200 | Air cool | | 125,000 | 135,000 | 15 | 50 |
| | Air | Rapid | 24 | 1300 | Furnace cool | 1 | 1200 | Air cool | | 125,000 | 135,000 | 15 | 50 |
| | Air | Rapid | 1 | 1400 | Furnace cool | 1 | 1200 | Air cool | | 125,000 | 135,000 | 15 | 50 |
| | Air | Rapid | 4 | 1400 | Furnace cool | 1 | 1200 | Air cool | | 125,000 | 135,000 | 15 | 50 |
| | Air | Rapid | 1 | 1500 | Furnace cool | 1 | 1200 | Air cool | | 125,000 | 135,000 | 15 | 50 |
| | Air | Rapid | 4 | 1500 | Furnace cool | 1 | 1200 | Air cool | | 125,000 | 135,000 | 15 | 50 |
| Isothermal equilibration anneal followed by furnace cooling to stabilizing temperature | Vacuum | With furnace | 4 | 1300 | Furnace cool | 1 | 1200 | With furnace | | 125,000 | 135,000 | 15 | 50 |
| | Vacuum | With furnace | 24 | 1300 | Furnace cool | 1 | 1200 | With furnace | | 125,000 | 135,000 | 15 | 50 |
| | Vacuum | With furnace | 4 | 1400 | Furnace cool | 1 | 1200 | With furnace | | 125,000 | 135,000 | 15 | 50 |
| | Vacuum | With furnace | 24 | 1400 | Furnace cool | 1 | 1200 | With furnace | | 125,000 | 135,000 | 15 | 50 |
| | Vacuum | With furnace | 4 | 1500 | Furnace cool | 1 | 1200 | With furnace | | 125,000 | 135,000 | 15 | 50 |
| | Vacuum | With furnace | 24 | 1500 | Furnace cool | 1 | 1200 | With furnace | | 125,000 | 135,000 | 15 | 50 |
| Isothermal equilibration anneal followed by step cooling to stabilizing temperature | Air | Rapid | 2 | 1300 | Furnace cool | 4 | 1200 | Air cool | | 125,000 | 135,000 | 15 | 50 |
| | Air | Rapid | 1 | 1400 | Step cool[4] | 4 | 1200 | Air cool | | 125,000 | 135,000 | 15 | 50 |
| | Air | Rapid | 1 | 1500 | Step cool[4] | 4 | 1200 | Air cool | | 125,000 | 135,000 | 15 | 50 |
| Isothermal equilibration anneal followed by isothermal stabilization anneal, using two furnaces | Air | Rapid | 4 | 1300 | Furnace quench | 4 | 1200 | Air cool | | 125,000 | 135,000 | 15 | 50 |
| | Air | Rapid | 4 | 1400 | Furnace quench | 4 | 1200 | Air cool | | 125,000 | 135,000 | 15 | 50 |
| | Air | Rapid | 4 | 1500 | Furnace quench | 4 | 1200 | Air cool | | 125,000 | 135,000 | 15 | 50 |
| Beta-field treatment | Air | Rapid | 1 | 1650 | Step cool[5] | 4 | 1200 | Air cool | | 125,000 | 135,000 | 15 | 50 |
| | Air | Rapid | 1 | 1650 | Step cool[6] | 4 | 1200 | Air cool | | 125,000 | 135,000 | 15 | 50 |
| | Air | Rapid | 1 | 1650 | Step cool[6] | 4 | 1200 | Air cool | | 125,000 | 135,000 | 15 | 50 |
| | Air | Rapid | 1 | 1650 | Furnace cool | 4 | 1200 | Air cool | | 125,000 | 135,000 | 15 | 50 |
| **RC-130B Wheel** | | | | | | | | | | | | | |
| As received | — | — | — | — | — | — | — | — | | 135,000 | 145,000 | 15 | 50 |
| Stabilization anneal | Air | With furnace | — | — | — | 24 | 1200 | With furnace | | 135,000 | 145,000 | 15 | 50 |
| | Vacuum | With furnace | — | — | — | 24 | 1200 | With furnace | | 135,000 | 145,000 | 15 | 50 |
| Isothermal equilibration anneal followed by furnace cooling to stabilizing temperature | Air | With furnace | 4 | 1400 | Furnace cool | 4 | 1200 | With furnace | | 125,000 | 135,000 | 15 | 50 |
| | Air | With furnace | 4 | 1500 | Furnace cool | 4 | 1200 | With furnace | | 125,000 | 135,000 | 15 | 50 |
| | Air | With furnace | 1 | 1600 | Furnace cool | 4 | 1200 | With furnace | | 125,000 | 135,000 | 15 | 50 |
| | Air | With furnace | 4 | 1600 | Furnace cool | 4 | 1200 | With furnace | | 125,000 | 135,000 | 15 | 50 |
| | Air | With furnace | 24 | 1600 | Furnace cool | 4 | 1200 | With furnace | | 125,000 | 135,000 | 15 | 50 |
| Isothermal equilibration anneal followed by furnace cooling to stabilizing temperature | Vacuum | With furnace | 4 | 1400 | Furnace cool | 4 | 1200 | With furnace | | 125,000 | 135,000 | 15 | 50 |
| | Vacuum | With furnace | 4 | 1500 | Furnace cool | 4 | 1200 | With furnace | | 125,000 | 135,000 | 15 | 50 |
| | Vacuum | With furnace | 1 | 1600 | Furnace cool | 4 | 1200 | With furnace | | 125,000 | 135,000 | 15 | 50 |
| | Vacuum | With furnace | 4 | 1600 | Furnace cool | 4 | 1200 | With furnace | | 125,000 | 135,000 | 15 | 50 |
| | Vacuum | With furnace | 24 | 1600 | Furnace cool | 4 | 1200 | With furnace | | 125,000 | 135,000 | 15 | 50 |
| Beta-field treatment | Vacuum | With furnace | 1 | 1710 | Furnace cool | 4 | 1200 | With furnace | | 125,000 | 135,000 | 15 | 50 |
| | Vacuum | With furnace | 1 | 1850 | Furnace cool | 4 | 1200 | With furnace | | 125,000 | 135,000 | 15 | 50 |
| Isothermal equilibration anneal followed by stabilizing anneal with varying intermediate cool | Vacuum | With furnace | 4 | 1600 | 7°F per min | 4 | 1200 | With furnace | | 125,000 | 135,000 | 15 | 50 |
| | Vacuum | With furnace | 4 | 1600 | Step cool[7] | 4 | 1200 | With furnace | | 125,000 | 135,000 | 15 | 50 |
| Isothermal equilibration anneal followed by varying stabilizing treatment | Vacuum | With furnace | 4 | 1600 | Furnace cool | 1 | 1200 | With furnace | | 125,000 | 135,000 | 15 | 50 |
| | Vacuum | With furnace | 4 | 1600 | Furnace cool | 37½ | 1200 | With furnace | | 125,000 | 135,000 | 15 | 50 |

[1] Bars were heated with the furnace (about 1½ hr to temperature) or by placing in a furnace already at temperature (rapid).
[2] Furnace-cooling rate was controlled at 2°F per min.
[3] Cooling rate in furnace was quite low, requiring 16 to 20 hr to reach room temperature from 1200°F.
[4] Furnace cooled 2°F per min to 1300°F, held 2 hr, furnace cooled 2°F per min to 1200°F.
[5] Furnace cooled 2°F per min to 1400°F, held 1 hr, furnace cooled 2°F per min to 1300°F, held 2 hr, furnace cooled 2°F per min to 1200°F.
[6] Furnace cooled 2°F per min to 1500°F, held 1 hr, furnace cooled 2°F per min to 1400°F, held 2 hr, furnace cooled 2°F per min to 1300°F, held 2 hr, furnace cooled 2°F per min to 1200°F.
[7] Furnace cooled 2°F per min to 1500°F, held 1 hr, furnace cooled 2°F per min to 1400°F, held 1 hr, furnace cooled 2°F per min to 1200°F.

Tangential test bars measuring approximately ½ in. sq and 3 in. long were cut from the rim section of the wheels for heat treatment. Triplicate Ti-140A bars, approximately 120° apart, were prepared. Duplicate RC-130B bars were prepared.

Vacuum heat treatments were conducted in an electrically heated tube furnace continuously pumped to maintain a vacuum of less than 1 $\mu$. Heat treatments in air were conducted in muffle-type electric furnaces with no attempt to control the atmosphere. Scaling in these treatments was negligible.

**Ti-140A Results:** An isothermal equilibration treatment below the beta transus followed by a water quench was included to show effects of increased annealing time and temperature on solution of $\alpha$. Increasing annealing temperature considerably reduced the ductility as quenched, indicating extensive solution of $\alpha$. Increasing annealing time had a similar but less marked effect.

**Table II.** *(Continued)*

| Tensile Properties | | | | |
|---|---|---|---|---|
| Ultimate Tensile Strength, Psi | 0.2 Pct Offset Yield Strength, Psi | Elongation, Pct in 1xDiam | Reduction in Area, Pct | Hydrogen, Wt Pct |
| 146,800 | 133,100 | 12.0 | 12.7 | 0.0290 |
| 152,300 | 150,400 | 10.0 | 10.5 | — |
| 150,300 | 148,700 | 10.7 | 13.0 | — |
| 152,200 | 151,200 | 9.5 | 9.9 | — |
| 179,100 | 181,700 | 1.0 | 2.3 | — |
| 181,100 | 181,400 | 0.7 | 2.7 | — |
| 130,200 | — | 0.0 | 0.5 | — |
| — | — | — | — | — |
| 142,600 | 135,800 | 17.8 | 17.0 | — |
| 143,200 | 137,300 | 11.8 | 13.0 | — |
| 143,500 | 134,800 | 15.5 | 13.6 | — |
| 143,000 | 136,200 | 18.7 | 18.2 | — |
| 140,200 | 132,500 | 17.0 | 16.3 | — |
| 141,800 | 133,500 | 19.0 | 15.6 | — |
| 141,500 | 136,500 | 19.0 | 17.5 | — |
| 141,100 | 131,800 | 16.7 | 16.7 | 0.0166 |
| 138,400 | 127,400 | 14.3 | 14.9 | 0.0053 |
| 137,400 | 125,600 | 23.7 | 22.7 | 0.0132 |
| 139,000 | 127,100 | 18.8 | 19.4 | 0.0015 |
| 140,700 | 130,800 | 25.5 | 26.6 | 0.0110 |
| 140,500 | 128,500 | 18.2 | 17.8 | 0.0024 |
| 142,200 | 134,500 | 15.5 | 15.5 | — |
| 138,900 | 132,400 | 16.5 | 17.5 | — |
| 140,300 | 135,200 | 9.8 | 11.0 | — |
| 142,500 | 135,500 | 17.0 | 16.7 | — |
| 142,900 | 135,900 | 17.3 | 15.3 | — |
| 143,400 | 137,600 | 9.0 | 10.0 | — |
| — | — | — | — | — |
| 140,000 | 134,700 | 14.3 | 14.1 | — |
| 139,300 | 134,100 | 13.2 | 14.2 | — |
| 139,300 | 134,700 | 10.5 | 12.4 | — |
| 132,500 | 127,600 | 10.5 | 11.8 | — |
| — | — | — | — | — |
| 146,200 | 135,400 | 7.8 | 10.3 | 0.0060 |
| 144,900 | 135,800 | 5.2 | 6.0 | 0.0062 |
| 145,700 | 131,200 | 12.0 | 15.6 | 0.0066 |
| 145,800 | 132,200 | 11.0 | 12.0 | — |
| 144,800 | 130,800 | 11.2 | 16.4 | — |
| 144,200 | 132,000 | 12.8 | 19.2 | — |
| 145,200 | 134,000 | 11.2 | 15.9 | — |
| 141,600 | 133,000 | 14.5 | 22.8 | 0.0100 |
| 145,800 | 131,000 | 12.5 | 20.3 | — |
| 143,300 | 130,300 | 11.5 | 16.2 | — |
| 143,800 | 130,200 | 14.5 | 23.5 | — |
| 142,000 | 129,300 | 15.5 | 21.9 | 0.0008 |
| 140,100 | 125,800 | 14.8 | 22.8 | — |
| 140,900 | 129,900 | 13.0 | 21.0 | 0.0016 |
| 133,100 | 122,400 | 7.5 | 14.4 | 0.0005 |
| 141,700 | 128,400 | 15.0 | 21.5 | — |
| 139,500 | 125,400 | 13.5 | 23.8 | — |
| — | — | — | — | — |
| 143,200 | 129,800 | 13.5 | 26.2 | — |
| 141,100 | 128,000 | 13.8 | 18.6 | — |

[a] Furnace cooled 2°F per min to 1500°F, held 1 hr, furnace cooled 2°F per min to 1200°F.
[b] Furnace cooled 2°F per min to 1500°F, held 2 hr, furnace cooled 2°F per min to 1400°F, held 2 hr, furnace cooled 2°F per min to 1300°F, held 4 hr, furnace cooled 2°F per min to 1200°F.

If the equilibration anneal below the beta transus was followed by a slow cool to the stabilization temperature and a short stabilizing treatment, a considerable improvement in ductility resulted. This was true both for vacuum and air heat treatments, and this type of treatment promoted the development of an equilibrium distribution of fairly coarse $\alpha$ particles in a $\beta$ matrix. A typical example of this structure is seen in Fig. 1. The heat-treated structure did not differ appreciably from the as-received structure. The chief result of heat treatment by equilibration, furnace cooling, and stabilization was an increase in structural uniformity and a closer approach to equilibrium compositional relationships.

Increasing equilibration annealing temperature resulted in improved ductility as long as the beta transus was not exceeded, while increasing equilibration annealing time tended to lower tensile ductility slightly. Increased time during vacuum annealing also resulted in lower hydrogen contents, which, as will be shown later, is a most desirable effect.

Vacuum treatments resulted in increased ductility as compared with air treatments. On the basis of tensile properties alone, vacuum treatment would not be required, since the air heat treatment raised tensile ductility sufficiently to allow the Ti-140A wheel to pass the acceptance test.

Variations in the intermediate cooling rate, such as use of a very slow step cool or a relatively rapid furnace quench, resulted in less desirable tensile properties than those obtained when using a controlled furnace-cooling rate of 2°F per min.

Two of the four heat treatments involving an initial annealing treatment in the $\beta$ field resulted in improved properties over the as-received condition but were less desirable than the $\alpha$-$\beta$ equilibration-type heat treatment. This effect was probably due to formation of an acicular $\alpha$ configuration, which is inherently less ductile than the structure illustrated in Fig. 1. The other two heat treatments resulted in a loss in ductility. The maximum improvement in ductility resulted from a 1 to 4 hr equilibration anneal about 100°F below the beta transus, slowly cooling (2°F per min) to 1200°F, and stabilizing at 1200°F.

**RC-130B Results:** As in the case of Ti-140A, the maximum improvement in ductility resulted from the equilibration, furnace cool, stabilization heat treatment. Probably because of its acicular as-received structure, ductility of RC-130B after heat treatment was less than that of Ti-140A. The most likely explanation is that, in the RC-130B wheel, which had been forged in the all-$\beta$ field, an acicular $\alpha$ configuration was formed by air cooling and was not removed by subsequent heat treatment. A typical example of this structure, exhibiting large, interwoven $\alpha$ needles in a matrix of $\beta$, is shown in Fig. 2. Holden et al.[3] have shown that this type of structure in a Ti-Mn $\alpha$-$\beta$ alloy is considerably less ductile than an equiaxed $\alpha$-$\beta$ structure such as that shown in Fig. 1.

Heat treatments designed to show the effects of varying the cooling rate between equilibration and stabilization indicated, as in the case of Ti-140A, that an optimum cooling rate existed. Similarly, an optimum stabilization time was indicated by tests on samples heat treated identically, except for the stabilization time.

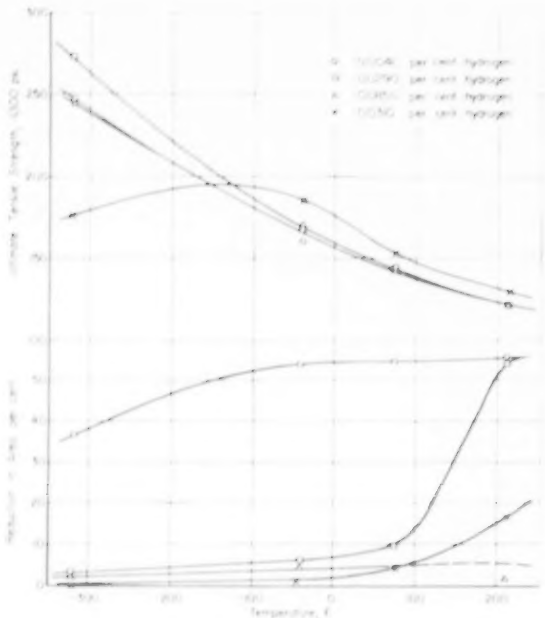

Fig. 4—Effect of hydrogen on the tensile properties of material from the Ti-140A compressor wheel

### Thermal Stability determined by tensile loss

The thermal stability of material from the forged wheels was determined by measuring the loss in room-temperature tensile properties occurring after exposure to the conditions listed in Table III.

Standard 0.125-in.-diam tensile specimens were used for tests at 600°F under stress. Standard 0.250-in.-diam specimens were used for unstressed tests. Special 0.250-in.-diam specimens, reduced slightly in overall length, were used for tests at 350°F under stress. Specimens exposed at 600° and 800°F were heated in a circulating-air pot furnace. A creep-test

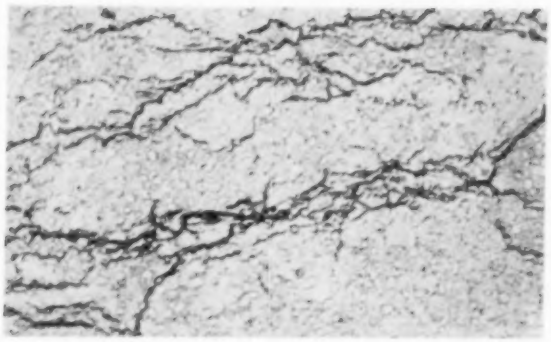

Fig. 5—Ti-140A containing 0.1310 wt pct H; dark phase believed to be hydride. Etched as in Fig. 1. X500

Table III. Thermal Stability Test Conditions

| Test No. | Temp, °F | Time, Hr | Applied Stress, Psi |
|---|---|---|---|
| 1 | 600 | 200 | None |
| 2 | 600 | 200 | 50,000 |
| 3 | 800 | 200 | None |
| 4 | 350 | 1000 | 50,000 |

apparatus was used to expose the 350°F test specimens.

The test specimens were machined to final dimensions before exposure. The small amount of scale formed during the test was removed by hand polishing with emery paper before testing.

**Ti-140A Results:** The thermal stability of material from the forged wheels was compared with material heat treated as follows: 1) equilibrated 4 hr at 1500°F, furnace cooled to 1200°F, and stabilized 1 hr at 1200°; heat treated in air; 2) same as above, but heat treated in vacuum. These heat treatments resulted in maximum improvements in ductility.

Average results of thermal-stability tests on the Ti-140A material are shown in Table IV. Both as-received samples and samples heat treated in air

Table IV. Thermal Stability of Heat-Treated Material From Both Compressor Wheels

| Heat Treatment and Test Condition | Ultimate Tensile Strength, Psi | 0.2 Pct Offset Yield Strength, Psi | Elongation, Pct in 4 D | Reduction in Area, Pct |
|---|---|---|---|---|
| Ti-140A | | | | |
| As received | | | | |
| No exposure | 138,800 | 128,400 | 10.4 | 12.0 |
| 1000 hr at 350°F, stressed | 140,800 | 133,600 | 9.5 | 11.2 |
| 200 hr at 600°F | 141,600 | 136,400 | 6.5 | 1.0 |
| 200 hr at 600°F, stressed | 147,900 | 141,200 | 2.0 | 3.1 |
| 200 hr at 800°F | 68,200 | — | 0.0 | 0.0 |
| Heat treated in air | | | | |
| No exposure | 136,800 | 128,200 | 23.2 | 25.4 |
| 1000 hr at 350°F, stressed | 138,800 | 130,800 | 10.8 | 11.2 |
| 200 hr at 600°F | 148,500 | 138,000 | 0.0 | 1.8 |
| 200 hr at 600°F, stressed | 144,000 | — | 0.0 | 0.8 |
| 200 hr at 800°F | 71,800 | — | 0.0 | 0.0 |
| Heat treated in vacuum | | | | |
| No exposure | 135,100 | 123,200 | 22.5 | 25.6 |
| 1000 hr at 350°F, stressed | 137,600 | 126,000 | 23.0 | 34.8 |
| 200 hr at 600°F | 137,600 | 123,400 | 13.0 | 13.6 |
| 200 hr at 600°F, stressed | 137,500 | 126,200 | 16.8 | 24.5 |
| 200 hr at 800°F | 144,400 | 125,600 | 12.0 | 13.2 |
| RC-130B | | | | |
| As received | | | | |
| No exposure | 146,200 | 135,400 | 7.8 | 10.3 |
| 1000 hr at 350°F, stressed | 145,200 | 131,200 | 7.5 | 13.4 |
| 200 hr at 800°F | 147,700 | 135,400 | 7.0 | 9.8 |
| Heat treated in air | | | | |
| No exposure | 141,600 | 133,000 | 14.5 | 22.8 |
| 1000 hr at 350°F, stressed | 144,600 | 131,600 | 11.2 | 15.3 |
| 200 hr at 800°F | 148,000 | 135,800 | 9.5 | 12.8 |
| Heat treated in vacuum | | | | |
| No exposure | 142,000 | 129,300 | 15.5 | 21.9 |
| 1000 hr at 350°F, stressed | 141,700 | 130,000 | 12.5 | 14.6 |
| 200 hr at 800°F | 144,300 | 131,200 | 10.8 | 15.4 |

Equilibrated below the beta transus, furnace cooled to the stabilizing temperature, and stabilized.

showed extreme embrittlement at 600° and 800°F, and moderate embrittlement at 350°F. The vacuum-heat-treated samples were ductile after exposure to all of the test conditions, with only a slight tendency toward embrittlement. The improved stability of vacuum-treated wheels was apparently due to the removal of hydrogen, which tends to accelerate the instability reaction. Air heat treatment slightly increased the tendency toward instability, possibly because of hydrogen pickup during heat treatment.

Results of tests carried out at 600°F under 50,000-psi stress did not vary appreciably from those of tests carried out at 600°F without stress. Measurements of gage length of the stressed samples before and after exposure indicated, within the limits of measurement—±0.5 pct—that no plastic strain occurred during exposure. Thus, it appears that the application of stress during exposure has little effect in thermal-stability testing unless a measurable plastic strain is produced.

Metallographic examination of as-received and air-equilibrated samples after exposure at 800°F showed a new phase distributed fairly extensively in the microstructure. An example of this phase is shown in Fig. 3. This phase was confined chiefly to the prior $\beta$-grain boundaries and appeared to form along the $\alpha$-$\beta$ interface. None of this phase was present in vacuum-annealed material. Tensile fractures in samples containing this phase occurred intergranularly along the prior $\beta$-grain boundaries. Some evidence of this phase was seen in the samples exposed at 600°F, but, in this case, it was not reproduced on repeated polishing and etching. Whether this phase was a hydride, a product of $\beta$ decomposition, or a hydrogen-induced etch effect could not be determined.

**RC-130B Results:** As-received material was compared with material heat treated as follows: 1) Equilibrated 24 hr at 1600°F, furnace cooled to 1200°F, and stabilized 4 hr at 1200°F. Heat treated in air; 2) Equilibrated 4 hr at 1600°F, furnace cooled to 1200°F, and stabilized 4 hr at 1200°F. Heat treated in vacuum. These were the most beneficial heat treatments for RC-130B.

Specimens from the RC-130B wheel were exposed only at 800° and 350°F. Average results of thermal-stability tests on RC-130B material are shown in Table IV.

The stability of all the material was good after exposure at 800° and 350°F, probably because of the low hydrogen content of the as-received wheel. In this case, vacuum treatment did not result in a noticeable difference from air heat treatment.

### Hydrogen-tolerance level is 0.004 pct.

Several tests were carried out to determine the effect of relatively high hydrogen content of the Ti-140A compressor wheel on properties known to be sensitive to hydrogen.[7] Four 0.25-in. diam rods were prepared containing fine, equiaxed $\alpha$-$\beta$ structures and hydrogen contents of 0.0040, 0.0290 (as received), 0.0850, and 0.1310 wt pct.

Five tensile specimens were prepared from material at each hydrogen level and tested with a head travel of 0.005 ipm at 212°, 75°, −40°, and −320°F. Two tests were made at room temperature and one at each of the other three temperatures.

Three notched tensile specimens, 1⅛ in. long with a 45° circumferential notch having a stress-concentration factor of three, were prepared from material at each hydrogen level. One of these specimens was tested in tension at room temperature with a head travel of 0.005 ipm. The remaining two specimens were tested in stress rupture at room temperature, using a constant-load test apparatus.

The results of these tests are plotted in Fig. 4. Only the vacuum-annealed material (0.0040 wt pct H) retained ductility below room temperature. Strength, on the other hand, showed relatively little variation with hydrogen content.

Results of notched tensile tests and stress-rupture tests conducted at room temperature are shown in Table V. Increased hydrogen content caused an almost progressive loss in notched tensile strength. Stress-rupture tests also indicated a rapid loss in properties with increased hydrogen content. All samples, except those containing 0.0040 wt pct H, failed in a brittle manner under a low applied stress or upon loading.

Metallographic examination showed evidence of a third phase in the microstructure of samples containing 0.0850 and 0.1310 wt pct H. The amount of this phase increased with hydrogen content. None of this phase was found in the as-received material, even though these samples showed definite indications of hydrogen embrittlement in mechanical tests. The loss of ductility at lower hydrogen contents is evidently promoted by some type of strain-aging mechanism. The third phase observed in the microstructure after testing occurred as a crazed pattern of long, thin, irregular plates along $\alpha$-$\beta$ interfaces. It was most pronounced near the surface of the specimens and often appeared to originate from a highly stressed region, such as the base of the threads. An example of the structure is shown in Fig. 5. Although the new phase was believed to be a hydride, it was not possible to make a definite identification.

**Table V. Effect of Hydrogen on Notched Tensile and Stress-Rupture Properties of the Ti-140A Compressor Wheel**

| Property | Hydrogen Content, Wt Pct | | | |
|---|---|---|---|---|
| | 0.0040 | 0.0290 | 0.0850 | 0.1310 |
| Notched tensile properties at 75°F | | | | |
| Ultimate tensile strength, psi | 214,000 | 208,000 | 185,000 | 157,000 |
| Reduction in area, pct | 7 | 5 | 3 | 2 |
| Notched stress-rupture properties at 75°F | | | | |
| Applied stress, psi | 200,000* | 120,000 | 120,000 | 120,000 |
| Rupture time, hr | 3.2 | <0.1 | <0.1 | <0.1 |
| Reduction in area, pct | 12.4 | 9.1 | 3.2 | 2.0 |
| Applied stress, psi | 200,000 | 110,000 | 80,000 | 60,000 |
| Rupture time, hr | <0.1 | 2.2 | 0.6 | 1.0 |
| Reduction in area, pct | 9.1 | 1.2 | 0.0 | 0.0 |

* Step loaded in 10,000-psi increments every 12 hr from 120,000 psi. Time to rupture measured from application of the final increment.

The amount of hydrogen present in the as-received Ti-140A compressor wheel was sufficient to induce brittleness in low-strain applications or at low temperatures. Vacuum annealing, which reduced hydrogen content to about 0.0040 wt pct, resulted in almost complete removal of the tendency toward embrittlement.

### Heat treatments caused appreciable improvement in tensile ductility of both Ti-140A and RC-130B forged compressor wheels.

Of the heat treatments examined, that in which the material was annealed about 100°F below the $\beta$ transus, slowly cooled to the stabilization temperature, and given a short stabilizing treatment

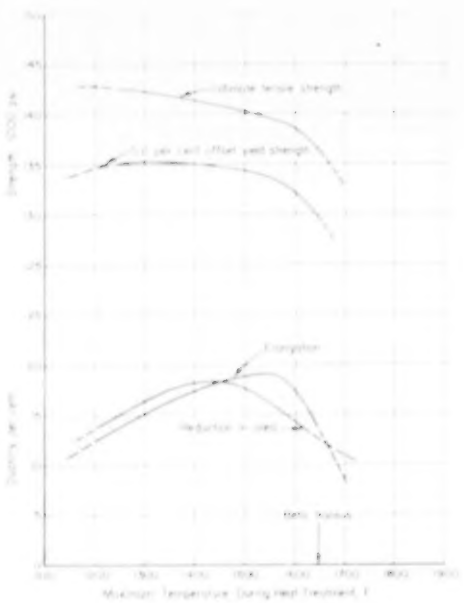

Fig. 6—Variation of tensile properties with the maximum temperature reached during heat treatment of Ti-140A, equilibrated, furnace cooled, and stabilized at 1200°F.

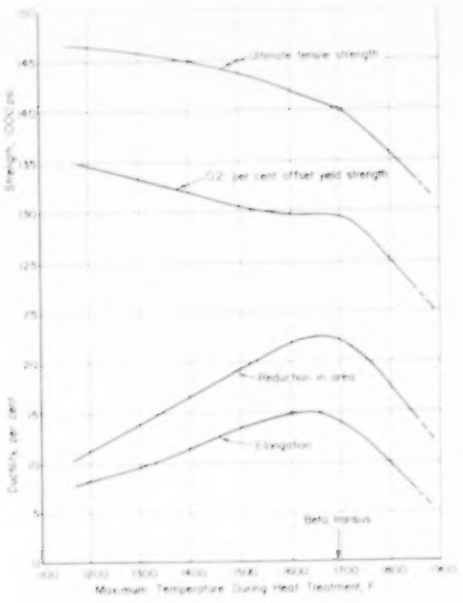

Fig. 7—Variation of tensile properties with maximum temperature reached during heat treatment of RC-130B, equilibrated, furnace cooled, and stabilized at 1200°F.

was most beneficial. During this treatment, the $\alpha$ phase is dissolved until only a relatively small amount remains to serve as nucleation sites; this is then reprecipitated at a rate sufficiently low to allow equilibrium to be maintained. Higher ductilities were developed in Ti-140A than in RC-130B by this treatment. This difference was probably related to the initial structure of the two alloys, since the RC-130B had an acicular $\alpha$ configuration which was not eliminated by heat treatment, while Ti-140A had an equiaxed $\alpha$ configuration. Vacuum heat treatments resulted in somewhat more ductile alloys than air heat treatments, but a slight difference in room-temperature tensile properties would not justify the expense of vacuum annealing.

Lenning et al,[3] in their study of the Ti-H system, reported a density decrease as hydrogen content was increased. This change in density with hydrogen content can be expressed as follows: Density, gm per cc = $-0.0071 \times$H content (atomic pct) + 4.5549. Since hydrogen is an interstitial solute in titanium, the observed decrease in density can be attributed to expansion of the titanium lattice. Thus, decreasing the hydrogen content from 0.0420 to 0 wt pct (roughly 2 atomic pct) by vacuum annealing would result in a contraction of the material of about 0.001 in. per in., assuming titanium to be isotropic. This secondary effect of vacuum heat treatment can be of considerable importance if finish-machined parts are to be heat treated.

The tensile properties of the heat-treated material were quite dependent on the maximum temperature reached during heat treatment, as is shown in Figs. 6 and 7. Both Ti-140A and RC-130B developed maximum ductility when the maximum temperature reached during heat treatment was 50° to 100°F below the $\beta$ transus. All samples represented in these figures were furnace cooled and stabilized following the high-temperature anneal.

Ductility first increased and then decreased as the maximum heat-treatment temperature increased; strength, on the other hand, decreased continuously, though at a much lower rate below the $\beta$ transus than above. These property changes observed can be explained qualitatively, if it is assumed that, during heat treatment, at least the following two reactions occur: 1) an increase in the coarseness of the $\alpha$-$\beta$ structure; and 2) an increase in the degree of attainment of equilibrium at the stabilizing temperature. The first reaction would lead to both decreased ductility and strength, effects of which would be much more severe when the heat treatment originated above the $\beta$ transus. The second reaction would lead to increased ductility and decreased strength, and would be most important below the $\beta$ transus.

Thermal-stability tests showed that stability was quite dependent on hydrogen content. Samples which were completely brittle when containing 0.0290 wt pct H showed almost no instability after vacuum heat treatment. This amount of hydrogen was also found sufficient to promote room-temperature strain-rate sensitivity, loss of ductility at low temperatures, and excessive notch sensitivity in Ti-140A. Again, vacuum heat treatment resulted in almost complete elimination of these tendencies. These results are in close agreement with those previously reported by Burte et al.[1]

Thus, it is apparent that vacuum heat treatment of high-hydrogen material, while not necessary to obtain satisfactory room-temperature properties, is highly desirable, since it improves elevated-temperature stability and low-temperature ductility and strain-rate and notch sensitivity.

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# Formation of Cracks in Soederberg Electrodes Used in Aluminum Reduction Plants

**Shrinkage and crack formation during baking of Soederberg anode paste for aluminum furnaces is discussed, and the authors demonstrate how this shrinkage may be reduced by applying suitable raw materials and paste compositions.**

by Ove Sandberg, Leif Olsen, and Torgrim Eftestoel

IN the vertical contact Soederberg electrode for aluminum furnaces more or less serious cracks are sometimes formed in the electrode, with harmful effect on furnace operation. The problem of crack formation has been studied on a laboratory scale to obtain a better understanding of this phenomenon.

The primary cause of crack formation is the shrinkage of the electrode carbon, which is opposed by the normal thermal expansion of the steel contact studs. The forces thus set up are active in the temperature interval from which the electrode obtains a rigid structure (450° to 500°C) to furnace operational temperature (950°C). A visualization of our reasoning is given in Fig. 1, which shows the different behavior of mild steel and electrode carbon during baking.

Research work performed to determine relationships between crack formation and shrinkage will be described. A brief description of the method developed for measurement of shrinkage will be given, as well as a critical analysis of this method. Also, raw material quality and paste composition variables will be related to shrinkage phenomena.

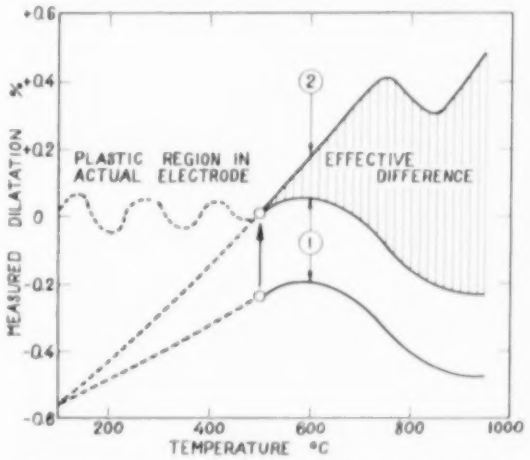


Fig. 1—Diagram showing different behavior upon heating of Soederberg anode and mild steel. Numbers refer to: 1, curve showing shrinkage of electrode during baking; and 2, curve showing dilatation of mild steel.

## Laboratory Test of Crack Formation

To test the tendency toward crack formations of Soederberg electrodes from different pastes, experiments were performed to simulate conditions of actual commercial electrodes, and are described as follows.

A cylindrical test electrode is prepared by tamping the electrode paste to be examined in a sheet iron casing, 350 mm diam by 700 mm height. The casing is perforated, with the holes (2 mm diam) 50 mm apart, to facilitate the escape of volatile matter during baking. A cylindrical steel stud of actual service size, 100 mm diam, is welded eccentrically to the bottom steel plate, and protrudes up through the paste. During baking, a crack may thus occur in the weaker part of the test electrode.

The electrode is baked by lowering the assembly slowly into an electric furnace, the temperature of which is kept constant at 900°C. After baking of the electrode, the sheet iron casing is removed and the electrode inspected for cracks.

The pictures in Fig. 2 are of three different pastes baked according to the present method. Electrode 1 has a large crack; electrode 2, a somewhat smaller one; and electrode 3, only a minor crack. As will be seen from the simplified sieve analysis diagram of Fig. 2, the compositions of the pastes in question are very different. This subject will be discussed later in greater detail.

## Crack Formation and Shrinkage Relationships

It is well known that shrinkage occurs in coke in the higher temperature ranges of coal carboniza-

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*The Transactions papers appearing in* JOURNAL OF METALS *during 1957 will subsequently appear in AIME Transactions Volume 209, and may be permanently referenced to that volume.*

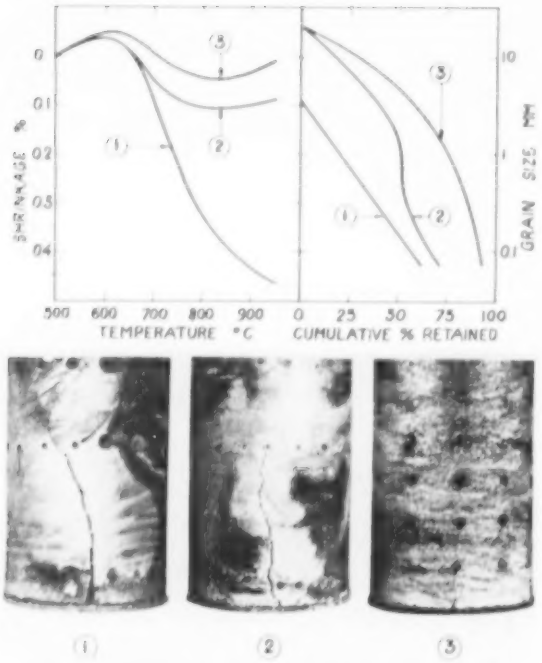


Fig. 2—Connection between crack formation and shrinkage. Ingot sections are from crack test of three different pastes. Shrinkage diagram is in upper left hand corner; simple diagram of the granular composition of the dry aggregate of the pastes, in upper right hand corner.

tion, and it is postulated that the prime reason for crack formations is a similar shrinkage of the electrode during baking. Therefore, dilatometric investigations of the shrinkage of electrodes during baking have been made. The temperature interval of interest for these investigations will be limited to the temperature at which the electrode obtains a rigid structure, and furnace operational temperatures.

Measurements were taken on specimens prepared from the pastes used for demonstration of crack formations. The pastes were prebaked to

Table I. Sieve Analyses of Dry Aggregate Coke for All Test Pastes Where Variations in Granulometric Composition Have Not Been Investigated

| U. S. Sieve No. | Opening, Mm | Cumulative Pct Retained |
|---|---|---|
| 4 | 4.76 | 3 |
| 8 | 2.38 | 14 |
| 16 | 1.19 | 27 |
| 30 | 0.59 | 42 |
| 50 | 0.297 | 54 |
| 100 | 0.149 | 67 |
| 200 | 0.074 | 80 |

500°C to obtain a rigid structure. From the coked paste, test specimens were prepared and measured in a special dilatometer, constructed for this very purpose. Results are presented in Fig. 2. Relationships between cracks and shrinkage are clearly demonstrated by comparing the pictures and the shrinkage diagram.

## Investigations of Shrinkage of Electrode Paste During Baking

**Description of the Method**—The method applied to shrinkage investigations involves the preparation of test specimens from a prebaked electrode sample, and dilatometric measurements on these specimens. The test specimens are prepared in the following manner: Raw paste is filled to a depth of 110 mm in a perforated sheet iron casing, 100 mm diam by 150 mm height. During baking, the paste is kept under a pressure of about 0.4 kg per sq cm. Temperature is raised from 20° to 500°C at a rate of 1°C per min, and held at 500°C for 2 hr, after which the furnace is cooled. From the resulting carbon cylinder, the specimens for shrinkage determinations are drilled out with a diamond core drill, after both ends have been turned in a lathe to give correct length. Finally, the specimen is longitudinally provided with a central hole, large enough to allow a protected thermocouple to protrude half way into it.

Shrinkage is then measured in the dilatometer, an assembly of four of which is shown in Fig. 3. A vertical cut of one dilatometer is shown in Fig. 4. It consists of a vertical tube furnace, in which the specimen is placed between parts of fused silica. The temperature is read by a thermocouple, the hot junction being placed in the center of the specially prepared specimen.

The temperature is raised at a uniform rate of 3°C per min. The expansion or contraction of the test specimen is transmitted by silica parts to a microgage, with a dial readable to 0.002 mm. The dilatometer is calibrated by measurement of a test specimen of fused silica. Since the coefficient of thermal expansion of fused silica is known, the expansion or contraction of the electrode specimen may be calculated by difference.

**Investigation of Experimental Variables**—Laboratory baking conditions are much different from

Table II. Sieve Analyses of Dry Aggregate Coke for Test Pastes Designed to Investigate Effect of Granulometric Composition*

| U. S. Sieve No. | Opening, Mm | Cumulative Pct Retained† | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
| ⅜ | 9.52 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 4 | 4.76 | 3 | 3 | 3 | 0 | 0 | 0 | 22 | 17 | 5 |
| 8 | 2.38 | 14 | 16 | 17 | 22 | 16 | 4 | 34 | 24 | 8 |
| 16 | 1.19 | 27 | 23 | 34 | 36 | 25 | 8 | 45 | 30 | 14 |
| 30 | 0.59 | 42 | 32 | 52 | 45 | 33 | 16 | 52 | 35 | 22 |
| 50 | 0.297 | 54 | 41 | 67 | 49 | 38 | 25 | 55 | 39 | 30 |
| 100 | 0.149 | 67 | 52 | 84 | 53 | 46 | 39 | 57 | 46 | 42 |
| 200 | 0.074 | 80 | 60 | 100 | 63 | 63 | 63 | 62 | 62 | 62 |

* Paste numbers refer to Figs. 8 and 9.
† Column numbers refer to different pastes. Paste No. 10 was the same as mixture 4; paste No. 11, the same as mixture 8.

those of electrode paste in commercial operation, where the time of baking is at least 100 times greater. The effect of prebaking time and temperature rise during measurement, therefore, had to be investigated in some detail.

Fig. 5, diagram *a*, demonstrates the effect of prebaking time on resulting shrinkage as shown by curve 2, 8 hr prebaking time (2 hr at 500°C); and curve 1, 24 hr prebaking time (18 hr at 500°C). Fig. 5, diagram *b*, demonstrates the effect of various upheat rates during measurement. Fig. 6 presents the effect of prolonged heating at 950°C upon shrinkage. It is evident from the preceding that the normal method of measurement does not yield total shrinkage.

**Raw Materials and Paste Compositional Variables**—The crack formation tests indicated a definite correlation between paste composition and shrinkage.

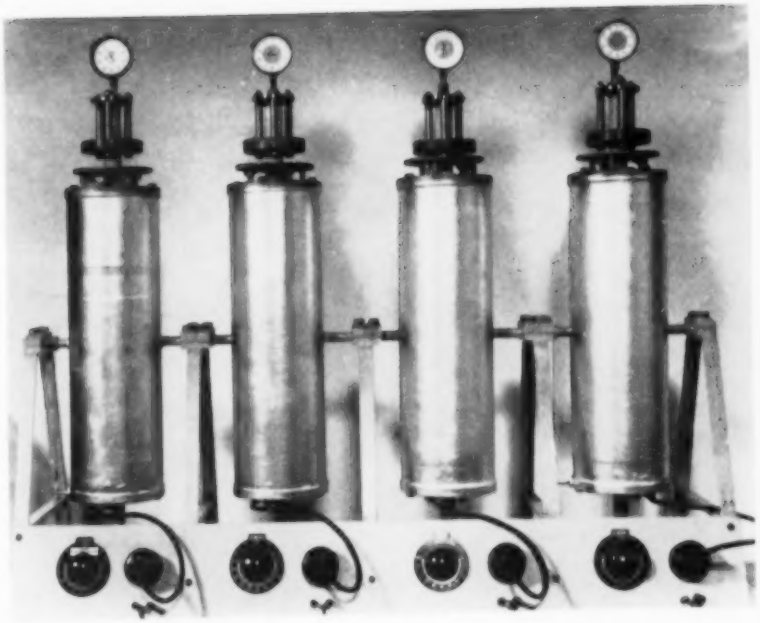

Fig. 3—Assembly of four dilatometers applied in the present work.

Systematic investigations have been carried out to test the effect of binder quality and content, coke quality (with special emphasis on the degree of calcining), and granulometric composition.

Investigations on raw materials and binder content were carried out with a constant granulometric composition of the test pastes, Table I. When granulometric composition was varied, binder quality and content were kept constant, a British coal tar pitch being employed at a concentration of 30 pct by weight. American petroleum coke was used for dry coke aggregate in the pastes.

*Effect of Binder Quality and Amount of Binder:* Three different binders, i.e., a British coal tar pitch, a vacuum pitch from Barrett,* and a petroleum pitch, were investigated. Differences in shrinkage were actually within the limits of experimental error. Binder content was varied from 20 to 40 pct by weight of the total paste, without any remarkable effect on shrinkage. Since binder content is kept within narrow limits in actual plant operation, this variable is not considered significant as far as variations in shrinkage are concerned.

* Barrett Div., Allied Chemical & Dye Corp., New York, N. Y.

*Degree of Calcination of Coke Aggregate:* The effect of degree of calcination of the coke aggregate on shrinkage is shown in Fig. 7. Calcining degree is measured in terms of real density, and the figure demonstrates clearly the marked effect of this variable. Shrinkage increases significantly with decreasing real density of the coke.

In Fig. 7, the curves are drawn for observations up to 1000°, while in all other series the curves end at 950°C. This demonstrates that curves 3 and 4 (and certainly 1 and 2) still exhibit, in the temperature interval 950° to 1000°C, a marked sloping tendency, while curve 5 has flattened out. This is the effect of prolonged heating at 950°C on shrinkage. As will be discussed later, only aggregate 5 consists of a dry aggregate coke with higher real density than the maximum obtained by heating raw petroleum coke to 950°C, which maximum real density has been determined to be 1.98 to 2.00 g per cu cm.

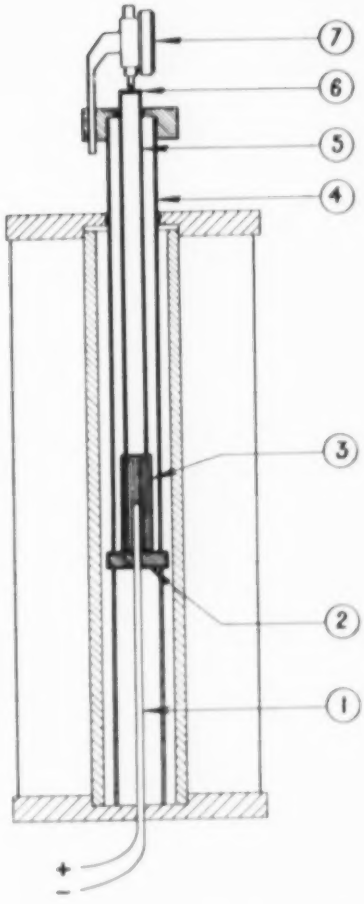


Fig. 4—Simplified vertical cut of the dilatometer, showing important parts. Numbers represent: 1, thermocouple; 2, fused silica disk; 3, test specimen 24 mm diam by 75 mm height; 4 and 5, fused silica tubes; 6, steel disk; and 7, microgage.

*Effect of Granulometric Composition:* Various tests were performed to investigate the effect of variations in coarse fractions (including maximum particle size), medium fractions, and fine fractions.

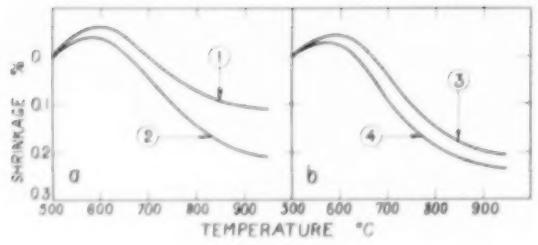


Fig. 5—Investigation of the conditions of measurement. Diagram *a* covers variations in prebaking time up to 500°C. Numbers refer to: 1, 24 hr prebaking time (18 hr at 500°C); and 2, 8 hr prebaking time (2 hr at 500°C). Diagram *b* shows results from variations in temperature rise during measurement. Numbers represent: 3, 3°C per min; and 4, 1°C per min.

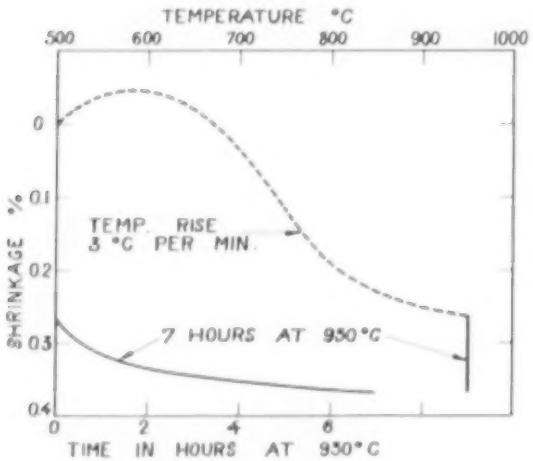


Fig. 6—Effect of prolonged measurement at 950°C on shrinkage. Upper curve (temperature abscissa) shows measured shrinkage up to 950°C, while lower curve (time abscissa) gives shrinkage at 950°C as a function of time.

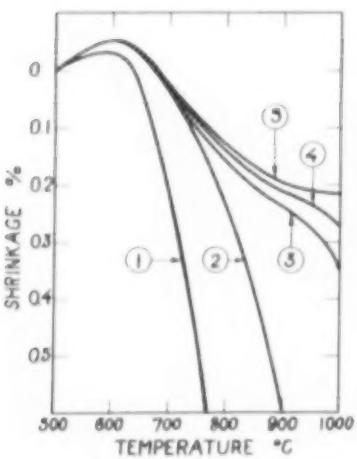


Fig. 7—Effect on shrinkage of calcining degree of dry aggregate coke. Numbers represent real densities of: 1, 1.65 g per cu cm; 2, 1.81 g per cu cm; 3, 1.89 g per cu cm; 4, 1.95 g per cu cm; 5, 2.05 g per cu cm.

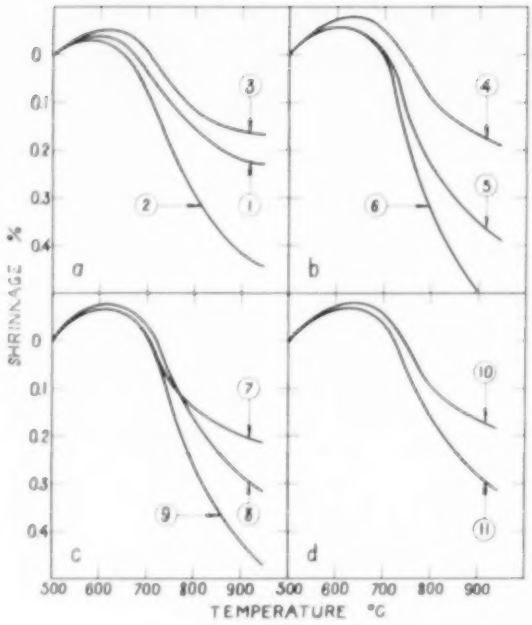


Fig. 8—Effect on shrinkage of granulometric composition of the dry aggregate. (For sieve analyses, compare with Fig. 9.) Diagram *a* demonstrates effect of variations in amount of fine fractions; diagrams *b* and *c*, effect of variations in proportion of coarse fractions to medium fractions. In diagram *d*, curve 10 is identical with curve 4 from diagram *b*, and curve 11 is identical with curve 8 from diagram *c*.

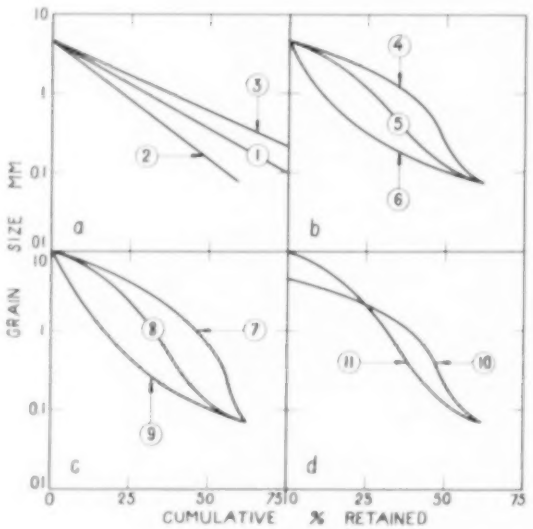


Fig. 9—Simplified sieve analysis diagrams of test pastes applied for investigation of variations in granulometric composition. Diagram *a* demonstrates effect of variations in amount of fine fractions; diagrams *b* and *c*, effect of variations in proportion of coarse fractions to medium fractions. In diagram *d*, curve 10 is identical with curve 4 from diagram *b*, and curve 11 is identical with curve 8 from diagram *c*.

Fig. 8, *a* through *d*, presents the shrinkage curves, while Fig. 9, *a* through *d*, gives the corresponding sieve analyses, which are also presented in Table II.

Fig. 8*a* shows mainly the effect of variations in the fine fraction. Fig. 8, *b* and *c*, shows separately the effect of changing the ratio of coarse fraction to medium fraction, with the fine fraction constant. Comparison of pastes of equal numbers in Fig. 8, *b* and *c*, gives an indication of the effect of maximum particle size on shrinkage, as all the former pastes have 5 mm maximum size and all the latter 10 mm maximum size. Fig. 8*d* gives an idea of the order of importance of large maximum size vs high ratio coarse to medium fraction.

For speculative reasons, which will be discussed later, a measurement on a mixture of only fine fraction and binder has been performed. Results of this measurement are given in Fig. 10 (curve 5) together with results from three different pastes (curves 4, 3, 2), a theoretical shrinkage curve (curve 6) based on a calculated shrinkage curve for the coked binder (curve 7), and the thermal expansion curve of dry aggregate coke (curve 1). The shrinkage curve of the coked binder is obtained by calculating contraction from real densities of binder coke calcined to different temperatures.

The theoretical paste shrinkage curve is computed by assuming a column of paste composed of 80 pct dry aggregate coke and 20 pct binder coke. The dry aggregate coke is assigned the normal thermal expansion (curve 1), while the binder coke on heating from 500° to 950°C follows curve 7 (both curves referring to total length of column). By algebraic addition of thermal expansion of dry aggregate coke and shrinkage of binder coke, the theoretical shrinkage is calculated as shown in curve 6.

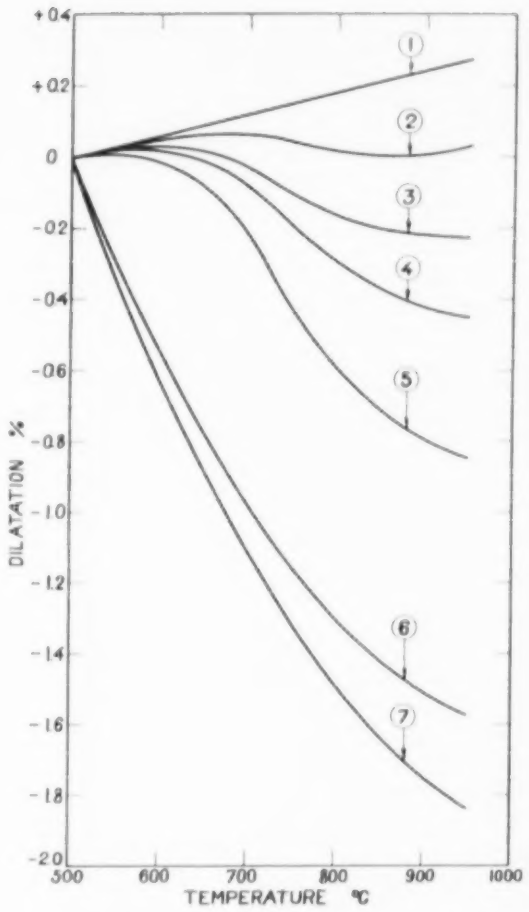


Fig. 10—Shrinkage curves drawn for demonstration of shrinkage mechanism. Numbers represent: 1, thermal expansion of coke; 2 through 4, pastes with decreasing amounts of coarser fractions; 5, mixture of milling product and binder; 6, theoretical shrinkage of anode; and 7, shrinkage of binder coke.

## Discussion

**Precision of Shrinkage Measurement**—The precision of the method is dependent on the preparation of test specimens and measurement of shrinkage in the same or in different dilatometers. If directions are closely followed, with special emphasis on temperature distribution and measurement, this experimental procedure gives a high degree of precision.

**Critical Considerations of the Method**—The comparison of crack tests and shrinkage indicates that dilatometric measurements are a suitable tool for research work in this field. Crack formations in actual furnace operations and shrinkage measurements on the electrodes in question also correlate well.

The true magnitude of electrode shrinkage in actual furnace operation will not be obtained by these measurements. Variations in prebaking time, temperature rise during measurement, and prolonged heating at 950°C indicate that higher shrinkage values should be expected in furnace operation than found by these measurements. Special attention is directed to the fact that prolonged heating at 950°C may have different effects on pastes of different quality, as has been especially demonstrated on pastes with varying degrees of calcination of the dry aggregate coke component. In such cases, laboratory measurements should also include prolonged heating at 950°C. As a means of comparing relative tendencies toward crack formations of different pastes, this method is considered suitable.

**Effects of Variations in Raw Materials**—The most important effect of variations in raw materials is that of the degree of calcination of the coke aggregate. The real density of a petroleum coke heat treated at 950°C reaches a maximum of 1.98 to 2.00 g per cu cm. Accordingly, a coke of this or higher densities will undergo no further shrinkage during furnace operation, and the total shrinkage may be attributed to the shrinkage of the binder coke.

Cokes of lower real density than 1.98 to 2.00 g per cu cm will, in the electrode of an aluminum furnace, shrink during the heating to 950°C. Electrodes with such cokes will contribute additional shrinkage, which may be far more severe than the shrinkage caused by the binder coke alone.

This point is emphasized, as there must be a compromise between the favorable effect of low calcined coke on electrode consumption, and the undesirable effect of serious crack formations.

**Effect of Granulometric Composition**—Variations in granulometric composition introduce complexities, since it is not possible to vary one fraction without also affecting the others. Even with this reservation, however, there seems to be a definite trend toward higher shrinkage with: 1) increasing amount of aggregate fine fraction (milling product); 2) decreasing ratio of coarse fractions (e.g., 10 to 2 mm particle size) to medium fractions (e.g., 2 to 0.2

mm); and 3) decreasing maximum size of coarse particles. The latter variation, within reasonable limits, gives the least pronounced effect.

While a decreasing amount of fine fraction has a favorable effect on the shrinkage of Soederberg electrodes, a rather high amount of this fraction will still be necessary to obtain the required flow properties in the final paste charged to the anode.

**Mechanism of Shrinkage**—To explain the effect of granulometric composition on shrinkage, Fig. 10 should be further considered. All the measured shrinkage curves show large deviations from the one theoretically calculated because of the assumption that the entire shrinkage of the binder coke is transmitted to the electrode.

Thus, it seems that the dry aggregate acts as a reinforcement in the electrode, counteracting the shrinkage of the binder coke. This reinforcement becomes greater the higher the percentage of coarse fraction contained in the paste and, most probably, the closer the packing of the dry aggregate particles. The paste mixture of fine fraction and binder curve (curve 5) in Fig. 10 comes closest to the theoretical curve 6, and it is quite evident that shrinkage tends to decrease with increasing amount of coarse particles in the paste.

If, however, a dry aggregate coke is used which itself will shrink with prolonged heat treatment in the furnace, the aggregate reinforcement action will certainly be reduced.

### Summary of Results

The correlation between crack formation and shrinkage of the Soederberg electrode during baking has been established by laboratory experiments.

Laboratory experiments point to the fact that electrode shrinkage during baking may be minimized by selecting a suitable granulometric composition of the coke aggregate component in the Soederberg paste.

**Discussion** of this paper sent (2 copies) to AIME by May 1, 1957 will appear in AIME *Transactions* Vol. 209, 1957, and in JOURNAL OF METALS, October 1957.

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# Equilibrium Between Titanium Metal, Titanium Dichloride, and Titanium Trichloride In Molten Sodium Chloride-Strontium Chloride Melts

**The equilibrium which exists between titanium metal, $Ti^{+2}$, and $Ti^{+3}$ in fused sodium chloride-strontium chloride baths has been determined. The effects of total titanium concentration, temperature, and the ratio of strontium to sodium chloride have been studied. In order to conduct the investigation a method for determining $Ti^{+3}$ was developed.**

by S. Mellgren and W. Opie

AN equilibrium state of importance in the process of electrolytically depositing titanium from fused chloride electrolytes is that which exists between titanium metal, titanium dichloride, and titanium trichloride. This can be expressed by the reaction

$$3\ TiCl_3 \rightarrow 2\ TiCl_4 + Ti^{+2} \qquad [1]$$

An investigation was undertaken to determine this equilibrium and to show the effect upon it of titanium concentration of the solution, temperature, and the proportion of strontium chloride and sodium chloride in the solution.

### Procedure

The general procedure used consisted of the following steps:

1) Development of an analytical method for determining $Ti^{+3}$ and total titanium.

2) Preparation of batches of fused salt solutions confaining various amounts of total titanium, $Ti^{+3}$, and $Ti^{+2}$.

3) Blending of the prepared batches and excess titanium metal in Vycor tubes and allowing the solutions to come to equilibrium. By blending, solutions with different total titanium content could be prepared and equilibrium could be approached from the high $Ti^{+3}$ and low $Ti^{+3}$ sides.

4) Studying the effect of titanium concentration, temperature, and the mole ratio of the solvent chlorides, SrCl, and NaCl, upon the equilibrium.

* The disproportionation equilibrium

$$2\ TiCl_3 \rightarrow TiCl_4 + Ti$$

is neglected in this study since it is assumed that the quantity of $Ti^{+2}$ in the melt is small. The reason is that the same amount of total titanium is found with or without zinc amalgam reduction of the solution of salt containing $Ti^{+3}$ and $Ti^{+2}$. If any $Ti^{+2}$ existed, the zinc amalgam reduction would be expected to give a higher value for total titanium in the melt.

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### Analysis for Titanium Dichloride

The method developed for determination of titanium dichloride is based on the fact that $TiCl_2$ will evolve hydrogen quantitatively from a weak acid solution according to the reaction

$$TiCl_2 + HCl \rightarrow TiCl_3 + \tfrac{1}{2} H_2 \qquad [2]$$

To measure the evolved gas an apparatus, shown in Fig. 1, was assembled. The 50 ml gas burette was calibrated from the upper stopcock in divisions of 0.1 ml. The side leveling tube is needed to bring the gas volume to atmospheric pressure when the readings are taken.

Analyses were made as follows:

1) Samples were taken from the salt melts by drawing the melt up into a piece of Pyrex tubing. A small amount of silica wool was placed in the tube end and the molten salt drawn through it to filter out any metal particles.

2) The 400 ml beaker shown in Fig. 1 was filled with dilute hydrochloric acid. Then, by means of a vacuum line, the acid was drawn up, filling the burette to the upper stopcock. The stopcock was then closed.

Since $H_2$ is soluble in acid-water solutions (up to 2 ml $H_2$ per 100 ml of solution at room temperature), the acid solution had to be saturated with hydrogen before using. This was accomplished by storing the acid in the presence of aluminum sticks. Hydrogen evolves and saturates the solution.

It is necessary to keep the acid concentration low because finely divided titanium, if present in the salt sample being analyzed, will also evolve hydrogen. The rate of evolution is very slow, however, compared to the rate of evolution due to titanium dichloride. A 50 to 1 volume ratio of $H_2O$ to HCl (specific gravity: 1.19) solution was found to be satisfactory.

3) Enough of the sample salt sticks were weighed to give a gas volume of from 20 to 40 ml.

Table I. Composition of Batches of Reduced Chlorides Prepared in Fused SrCl$_2$-NaCl Eutectic Melts

| Batch No. | Wt Pct $Ti^{+2}$ | Wt Pct $Ti^{+3}$ | Wt Pct Total Ti | Pct $Ti^{+3}$ | Remarks |
|---|---|---|---|---|---|
| 1 | 0.78 | 0.43 | 1.21 | 65 | No metal deposition |
| 2 | 1.76 | 0.24 | 2.00 | 88 | Some metal deposition |
| 3* | 2.45 | 0.00 | 2.45 | 100 | Much metal deposition |
| 4 | 1.54 | 5.06 | 6.60 | 23 | No metal deposition |
| 5 | 1.31 | 6.31 | 7.62 | 17 | No metal deposition |

* Batch No. 3 was produced using 8 faradays per mole of tetrachloride introduced. The diaphragm was quickly removed from the cell and chilled.

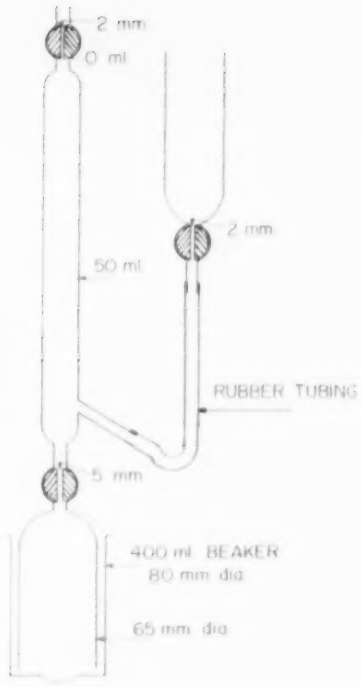


Fig. 1—Hydrogen evolution apparatus for determining $Ti^{+2}$.

These were introduced between the 400 ml beaker and the bell and maneuvered into the center of the bell as quickly as possible. The evolved gas displaces the acid in the burette. It was found that the salt samples could be stored for as long as two years without changing if the ends of the glass tubes holding them were sealed with rubber stoppers.

4) After evolution was complete the leveling tube was used to obtain a volume reading and the percentage titanium dichloride calculated using the equation

$$\% Ti^{+2} = \frac{X \times P \times 2 \times 47.9 \times 273 \times 100}{T \times 22410 \times 760 \times W}$$

where $X$ equals ml of displaced volume in the gas burette at atmospheric pressure; $P$, pressure in mm of evolved hydrogen (corrected barometric pressure minus the vapor pressure of water at the temperature of measurement); $T$, temperature in °K; and $W$, weight of the sample in grams.

### Analysis for Titanium Trichloride

By dissolving the salt samples in dilute HCl under an inert atmosphere and titrating directly with

Table II. Effect of Total Titanium Content upon the Titanium Metal, $Ti^{+2}$, $Ti^{+3}$ Equilibrium in a Fused Solution of 3 Mole NaCl to 2 Mole SrCl$_2$ at 700°C

| Experiment No. | Total Titanium, Pct | $\frac{Ti^{+2}}{Ti^{+2} + Ti^{+3}} \times 100$ at Start | Composition at Equilibrium, Pct $Ti^{+2}$ | Composition at Equilibrium, Pct $Ti^{+3}$ | $\frac{Ti^{+2}}{Ti^{+2} + Ti^{+3}} \times 100$ at Equilibrium | No. of Analyses at Equilibrium | Variation Range, Pct |
|---|---|---|---|---|---|---|---|
| 1 | 1.68 | 88 | 1.41 | 0.27 | 84 | 2 | ±0.5 |
| 2* | 2.45 | 100 | 2.03 | 0.42 | 83 | 2 | ±1.0 |
| 3 | 3.96 | 58 | 3.24 | 0.72 | 82 | 5 | ±2.5 |
| 4 | 4.39 | 50 | 3.58 | 0.80 | 82 | 3 | ±2.0 |
| 5 | 4.95 | 40 | 4.03 | 0.92 | 81 | 4 | ±2.0 |

* If finely divided metal is used, the equilibrium is reached as soon as the salt is fused. If coarse Kroll sponge is used, 2 to 3 hr may elapse before equilibrium is reached.

standard ferric ammonium sulfate using an ammonium thiocyanate indicator the total titanium can be determined without reduction of the solution by zinc amalgam. Trivalent values were obtained by subtracting the hydrogen evolution divalent results from the total titanium. The direct titration method was checked against the standard zinc amalgam method for determining total titanium.

### Sample Preparation

Samples of reduced titanium chlorides were prepared in an electrolytic cell using a 2 in. ID Norton RA98 Alundum diaphragm $\frac{3}{8}$ in. thick and 6 in. deep as a cathode chamber. A graphite plate served as an anode and was located in the salt bath outside of the diaphragm. A eutectic melt of strontium chloride-sodium chloride was used as the electrolyte and titanium tetrachloride was introduced into the cathode chamber through a hollow nickel cathode at a temperature of about 700°C. The cell was sealed against air contamination. By varying the ratio of the tetrachloride introduction rate to the direct current the compositions shown in Table I were made.

### Equilibrium Results

From the salt batches listed in Table I a number of melts were made up containing various amounts of total titanium with excess titanium metal, introduced either as fine Kroll sponge or metal from batch 3, Table I. The compositions were sealed in Vycor tubes and maintained at constant temperature by submerging the tubes in a molten salt bath. The temperature of the bath could be kept within ±2°C. A static argon atmosphere was kept over the salt melts in the tubes. The melts were sampled periodically and analyzed until constant results were attained indicating an equilibrium condition.

### Effect of Titanium Concentration

Table II contains data obtained at 700°C in a melt containing 3 mole NaCl to 2 mole $SrCl_2$. The total titanium content was varied between 1.68 and 4.95 wt pct. The equilibrium value was approached from the low dichloride side in runs 3 to 5 and from the high dichloride side in runs 1 and 2.

Table III. Effect of Temperature upon the Ti, $Ti^{++}$, $Ti^{+3}$ Equilibrium in a Fused Solution of 3 Mole NaCl to 2 Mole $SrCl_2$ containing 4.3 Wt Pct Total Titanium

| Temperature, °C | $\frac{Ti^{+3}}{Ti^{++} + Ti^{+3}} \times 100$, Pct |
|---|---|
| 650 | 82 to 83 |
| 700 | 79 to 83 |
| 800 | 80 to 85 |
| 850 | 83 to 86 |

There may be a slight tendency for the equilibrium value of $Ti^{+3}$ to increase with decreasing total titanium content, but the difference noted was within analytical experimental error.

### Effect of Temperature

The change in the equilibrium value in the temperature range 650° to 850°C was found to be within the expected error of the analytical method, Table III.

### Effect of Varying the NaCl to $SrCl_2$ Ratio

There was reason to believe that the equilibrium between $TiCl_3$, $TiCl_4$, and titanium metal depends on the relative amounts of NaCl and $SrCl_2$ in the salt melt. The system, $SrCl_2$, NaCl, $TiCl_3$, $TiCl_4$ and titanium metal, is determined by three phases and four components. Using the phase rule, the number of degrees of freedom existing is three, $F = C - P + 2 = 3$.

If the temperature and the pressure are fixed there is still one degree of freedom which can be eliminated by fixing a ratio such as NaCl/$SrCl_2$. The amount of $TiCl_3$ and $TiCl_4$ that can exist in equilibrium thus depends on the above ratio. To study this effect melts were made up in such a manner that the final total reduced titanium in the salt melt was between 2.5 and 3.0 pct. The $SrCl_2$/NaCl mole ratio was varied from 20/80 to 80/20. Data are included in Table IV. In the two extreme cases the temperature was varied between 700° and 850°C with no appreciable effect noted.

### Solution Ideality

From Table II the mole fractions of $TiCl_3$ and $TiCl_4$, assuming Eq. 1, existing at equilibrium for each experiment have been calculated and are listed in Table V. Calculations were based on 40 mole pct $SrCl_2$-60 mole pct NaCl as the solvent and it is assumed that this ratio did not change during the runs.

Writing the equilibrium equation in the following way

$$3\,TiCl_3\ (\text{in solution}) \rightleftharpoons 2\,TiCl_4\ (\text{in solution}) + Ti\ (\text{solid}) \quad [4]$$

the equilibrium constant will be

$$K = \frac{(N_{TiCl_4})^2}{(N_{TiCl_3})^3}. \quad [5]$$

If the solution is ideal, the ratio $(N_{TiCl_4})^2/(N_{TiCl_3})^3$ would be constant when the mole fractions listed in Table V are substituted in Eq. 5. Such is not the case. Table VI shows that the calculated $K$ values change as the total titanium in the salt melt varies.

### Discussion of Results

No change was noted in the temperature range between 650° and 800°C. It is estimated that the

Table IV. Effect of $SrCl_2$ to NaCl Mole Ratio on the Ti, $Ti^{++}$, $Ti^{+3}$ Equilibrium

| Ratio, Mole Pct $SrCl_2$ / Mole Pct NaCl | Reduced Titanium at Equilibrium, Pct | $Ti^{+3}$ of Reduced Titanium at Start, Pct | $Ti^{+3}$ of Reduced Titanium at Equilibrium, Pct | No. of Analyses at Equilibrium | Variations, Pct | Temperature, °C |
|---|---|---|---|---|---|---|
| 20/80 | 2.5 | 20 | 78 | 6 | ±0.3 | 700 to 850 |
| 40/60* | 3.96 | 38 | 82 | 5 | ±2.5 | 700 |
| 40/60* | 2.45 | 100 | 83 | 2 | ±1.6 | 700 |
| 80/20 | 3.0 | 20 | 93 | 5 | ±1.5 | 700 to 850 |

* From Table II.

Table V. Concentration of Titanium and Mole Fractions of $TiCl_2$ and $TiCl_3$ in the Melt

| Experiment No. | Total Titanium, Pct | $N_{TiCl_2}$, Mole Fraction $TiCl_2$ | $N_{TiCl_3}$, Mole Fraction $TiCl_3$ |
|---|---|---|---|
| 1 | 1.68 | $3.23\times10^{-3}$ | $6.15\times10^{-6}$ |
| 2 | 2.45 | $4.56\times10^{-3}$ | $9.68\times10^{-6}$ |
| 3 | 3.96 | $7.44\times10^{-3}$ | $16.57\times10^{-6}$ |
| 4 | 4.38 | $8.28\times10^{-3}$ | $18.80\times10^{-6}$ |
| 5 | 4.89 | $9.33\times10^{-3}$ | $21.20\times10^{-6}$ |

Table VI. Concentration of Titanium in the Melt and Exponential Ratios of Mole Fractions of $TiCl_2$ and $TiCl_3$

| Experiment No. | Total Titanium, Pct | $K = \frac{(N_{TiCl_2})^3}{(N_{TiCl_3})^2}$ |
|---|---|---|
| 1 | 1.68 | 1.21 |
| 2 | 2.45 | 0.99 |
| 3 | 3.96 | 0.67 |
| 4 | 4.38 | 0.63 |
| 5 | 4.89 | 0.56 |

analytical error can be as high as 5 pct. Therefore if the equilibrium is affected by an amount less than this by varying the temperature within this range it could not be noted.

There is a definite trend for the ratio $(N_{TiCl_3})^2/(N_{TiCl_2})^3$ to decrease with increasing titanium concentration in the salt melt, although this variation is not great, Table VI. Because of the changing value of $K$ it must be concluded that the solution is not ideal considering Eq. 1.

If the divalent and trivalent titanium chlorides are converted to mole fractions assuming complex formation such as

$$2\ TiCl_3 \rightleftarrows Ti_2Cl_6 \text{ and } 2\ TiCl_2 \rightleftarrows Ti_2Cl_4$$

and

$$TiCl_2 \cdot Ti_2Cl_4 \rightleftarrows TiCl_3 \cdot \tfrac{1}{2}\ Ti_2Cl_6 + Ti$$

a constant value for $K$ may be obtained.

## Summary

It has been demonstrated that an equilibrium exists between divalent, trivalent, and metallic titanium in a fused chloride system using sodium and strontium chlorides as solvents. This equilibrium is unaffected by temperature change between 650° and 800°C (within analytical error range of 5 pct) or by varying the total titanium concentration between 1.7 and 5.0 pct total titanium in solution. It is markedly affected by varying the $SrCl_2$ to NaCl mole ratio of the solvent, the divalent titanium at equilibrium decreasing with increasing NaCl content. The solutions studied are not ideal.

## Acknowledgment

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# Roasting Reaction of Ferrous Sulfide

**The rate and mechanism of oxidation of ferrous sulfide have been studied by means of the spring balance and X-ray diffraction analysis over the temperature range from 500° to 700°C. The complete oxidation to oxides begins at 600°C. At the initial stage of oxidation, a slight increase of weight was found. It was observed by X-ray that the deficiency of iron ion in FeS occurs in this stage. This is interpreted as follows: iron ion reacts with oxygen by migrating from the interior of FeS crystal to the surface, without evolution of $SO_2$, until the deficiency of iron attains to a limiting value. Above 600°C the rate of oxidation does not change with temperature, so the rate-determining factor is diffusion of gas. Since the observed rate of oxidation depends upon the amount of the sample, the diffusion in the bed of the sample may determine the rate.**

by Kichizo Niwa, Tsuguyasu Wada, and Yutaka Shiraishi

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ALTHOUGH studies on the roasting of ferrous sulfide have been carried out by several investigators, some disagreements still remain among those results. Schwab and Philinis[1] have measured the rate of oxidation by a precise thermobalance and have concluded that the rate is controlled by diffu-

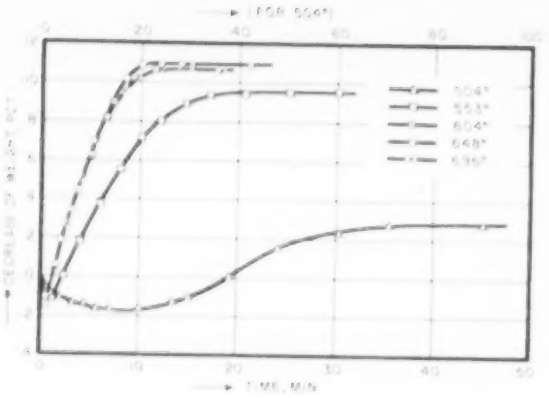

Fig. 1—Change in weight during oxidation of ferrous sulfide.

sion of gas, similar to the case of pyrite. Chufarov and Averbukh[3] have found that the oxidation of ferrous sulfide proceeds as follows: Up to 250°C the oxidation takes place without evolution of gas, showing that the product is $FeSO_4$. At 350° to 450°C the product is a mixture of $FeSO_4$ and iron oxides, and above 450°C it is a mixture of $Fe_2O_3$ and $Fe_3O_4$. Kameda, Yazawa, and Kurosawa[4] have investigated the kinetics of the oxidation of artificial ferrous sulfide, and have shown that the initial stage of the reaction is autocatalytic, an induction period being observed. According to Kushima and Kondo,[5] who have studied the oxidation products by a thermobalance, sulfate which is produced during oxidation is presumed to be $Fe_2(SO_4)_3$; also, complete formation of oxides is observed above 600°C.

Table I. An Example of X-Ray Powder Photographs*

| d (kX) | I | Substance |
|---|---|---|
| 5.72 | w | FeS (001) (?) |
| 2.97 | m | FeS (100) |
| 2.70 | s-w | $Fe_2O_3$ |
| 2.64 | st | FeS (101) |
| 2.52 | s | $Fe_2O_3$, $Fe_3O_4$ |
| 2.27 | v-w | $Fe_2O_3$ |
| 2.16 | v-w | FeO |
| 2.10 | s-w | $Fe_3O_4$ |
| 2.06 | st | FeS (102) |
| 1.840 | v-w | $Fe_2O_3$ |
| 1.717 | st | FeS (110) |
| 1.680 | v-w | $Fe_2O_3$ |
| 1.612 | v-w | $Fe_3O_4$ |
| 1.602 | w | FeS (103) |
| 1.483 | w | FeS (200) |
| 1.440 | w | FeS (201) |
| 1.426 | w | FeS (004) |
| 1.319 | m | FeS (202) |

* Sample: No. 7, 600°C, 100 sec.

In the present report, the kinetics of oxidation is studied by means of a spring balance, and the mechanism of reaction during the initial stage is discussed upon the basis of observations by the X-ray diffraction method.

Ferrous sulfide usually contains some excess sulfur, forming the so-called nonstoichiometric compound over the range of from $FeS_{1.00}$ to, as the most reliable value, $FeS_{1.09}$.[6] The formation of this nonstoichiometric compound is due to the escape of cations from their lattice sites to form the cation vacancies.[7,8] The crystal structure of FeS is the hexagonal of NiAs type, and the lattice constants $a$ and $c$ vary with the change of composition, such that the lattice contracts when the atomic fraction of sulfur is increased.[9,10] The change of composition is, therefore, detectable by X-ray diffraction.

## Experiments on Oxidation Process

The rate of oxidation of artificial ferrous sulfide in a stream of air was measured by a spring balance over the temperature range from 500° to 700°C.

**Experimental Method and Material:** The spring balance used was the same as that described in a previous paper.[3] Its sensitivity is about 0.3 mg per 0.1 mm elongation. The rate of flow of air was fixed at 200 cu cm per min, because no effect on the rate of reaction was observed when the rate of flow was increased above this.

Ferrous sulfide used was prepared as follows: Two boats which had been filled with electrolytic iron and sulfur, respectively, were put in a quartz tube, and after evacuation up to about $10^{-4}$ mm Hg, the tube was heated to 800° to 900°C. After the sulfurization had been finished, which took about two days, the product was powdered and was then evacuated again at 900°C to remove excess sulfur. The final product was powdered again and its fraction between 170 and 200 mesh was collected by sieving.

Table II. Products of Initial Stage Detected by X-Ray Diffraction

| Reaction Temperature, °C | Time, Sec | Detected Substances (Except FeS) |
|---|---|---|
| 500 | 30 | — |
| 500 | 75 | $Fe_2O_3$ |
| 500 | 120 | $Fe_2O_3$ |
| 500 | 180 | $Fe_2O_3$ |
| 500 | 240 | $Fe_2O_3$, ($Fe_3O_4$)* |
| 600 | 20 | — |
| 600 | 40 | $Fe_2O_3$ |
| 600 | 60 | $Fe_2O_3$ |
| 600 | 100 | $Fe_2O_3$, $Fe_3O_4$, (FeO) |
| 600 | 150 | $Fe_2O_3$, $Fe_3O_4$, (FeO) |

* The existence of substances in parentheses is not certain.

The analysis of sample resulted in 61.98 pct Fe and 38.11 pct S. These values were slightly different from those of the stoichiometric composition, 63.52 pct Fe and 36.48 pct S. The atomic percentage of sulfur was 51.71. Thus the sample remained nonstoichiometric, in spite of such a treatment in vacuum at elevated temperature. No other lines except those of ferrous sulfide were observed by X-ray powder photograph. In every run 0.1 g of sample was used.

**Results and Discussion:** Results of experiments are shown in Fig. 1, in which the coefficients of decrease of weight (pct) are plotted against reaction time (min).

At reaction temperature of 504°C, some increase of weight appears first, and continues for about 15 min until the weight begins to decrease gradually. (In this case the scale of abscissa in Fig. 1 is reduced to a half of others.) The decrease of weight, however, is only 2.8 pct even after 90 min, which is far less than that of oxides; viz., 9.1 pct for FeO, and 12.2 pct for $Fe_2O_3$. Thus, the sulfate may exist. At 553° small amounts of sulfate may also be formed;

Table III. Change of Lattice Constants and Composition of FeS During the Initial Reaction

| Specimen | Initial | | | Oxidized at 500°C | | | Oxidized at 600°C | | |
|---|---|---|---|---|---|---|---|---|---|
| | Lattice Constant, (kX) | Atomic Pct S by Ueda | Atomic Pct S by Haraldsen | Lattice Constant, (kX) | Atomic Pct S by Ueda | Atomic Pct S by Haraldsen | Lattice Constant, (kX) | Atomic Pct S by Ueda | Atomic Pct S by Haraldsen |
| A-axis | 3.44$_5$ | ~50 | 51.4 | 3.44$_5$ | ~50 | 52.0 | 3.43$_5$ | 52.7 | 52.5 |
| C-axis | 5.77$_2$ | 51.0 | 51.4 | 5.72$_2$ | 52.5 | 52.5 | 5.70$_4$ | 52.8 | 53.0 |
| Mean | — | 50 to 51 | 51.4 | — | 51 to 52 | 52.3 | — | 52.8 | 52.8 |

above 604°C, the product is undoubtedly oxides only.

It must be noticed, however, that these results were obtained under the condition of very large excess of the air flow. It is quite certain that a significant amount of iron sulfate will form in roasting at 500° to 600°C, if the roasting is done in a limited supply of air, as in practical fluid-bed roasting.

Two striking phenomena are observed from Fig. 1. First, for every temperature there is an increase of weight of about 1.0 to 1.8 pct at the initial stage of reaction. Secondly, above 604°C the rate of oxidation does not change with temperature. The latter means that the energy of activation for the reaction is very small, so the rate-determining factor may be diffusion of gas.

On these two points the discussions will be offered and the further experiments will be described in the following section.

## The Mechanism of Initial Reaction

An interesting problem is what causes the initial increase of weight. The amounts of increase are too great to regard the cause as adsorption of oxygen. The formation of sulfate is more probable. No reliable evidence has been published up to now, however, as to whether or not the formation of sulfate is an intermediate stage of oxidation to oxides.

The mechanism of the increase of weight will now be discussed on the basis of the results from X-ray diffraction analysis of oxidized samples.

**Experiments:** Samples were quenched in nitrogen gas in a way similar to that adopted in previous work,[7] and then analyzed by means of an X-ray powder photograph. In two cases of reaction temperature, 500° and 600°C, the change of weight was observed as shown in Fig. 2A, in which each point represents the prepared sample and its weight. Since 200 mg of sample was taken, the rate of oxidation was somewhat lower than that shown in Fig. 1. (The reason will be discussed later.) X-ray diffraction patterns were obtained by means of ordinary photographic methods, using a powder camera with diameter of 90 mm and CoK$\alpha$ radiation.

For the ordinary material, the minimum amount to be detectable by X-ray diffraction is somewhere between 0.5 and 5 pct by weight,[8] although it depends on the material in question. While the oxides of iron are relatively easy to detect, sulfates are supposed to be rather insensitive. Therefore, 5 pct or more sulfate may be necessary to obtain the distinct lines. These points are important when the results of X-ray diffraction are discussed.

**Results and Discussion:** Since all data of X-ray diffraction cannot be present on account of space limitation, only one example is shown in Table I. In this case $Fe_2O_3$ and $Fe_3O_4$ were identified, while the existence of FeO is not certain because the observed line is only a very weak one. Identified substances are summarized in Table II.

A striking phenomenon is the shift in lines of FeS, some of which are shown in Fig. 2B. This indicates a contraction of lattice constant of ferrous sulfide, which is probably due to the increase of the atomic fraction of sulfur in it.

This fact suggests that during the initial stage the iron ions in FeS combine with oxygen, forming iron oxides on the surface, but sulfur ions remains in FeS without formation of sulfur dioxide, resulting in the increase of atomic fraction of sulfur. Since the relation between the lattice constants and the atomic pct of sulfur have been reported,[9,10] it can be estimated what change of the composition corresponds to such a contraction of lattice. If it is assumed that the initial reaction is mainly proceeded as

$$FeS_{1+x} + \frac{2}{3}x\,O_2 = Fe_{1-x}S_{1+x} + \frac{x}{3}Fe_2O_3 \qquad [1]$$

it can be determined what increase of weight is to be expected for such a change of composition, or $x$, in the initial stage. The calculation and the comparison with the experimental values are done as follows.

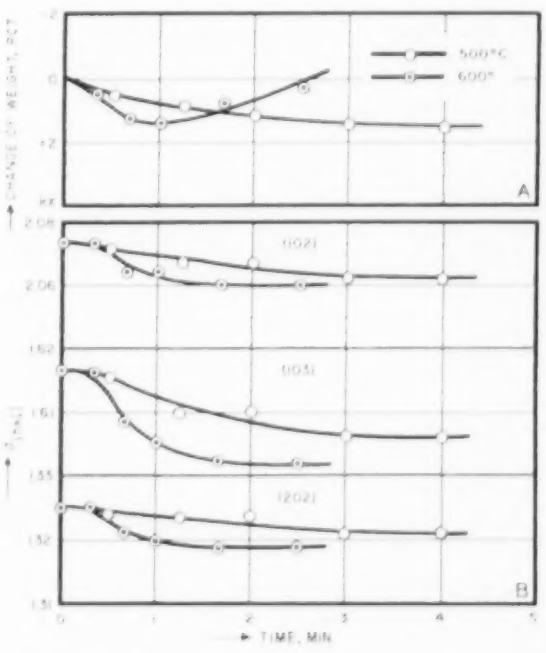


Fig. 2—Reaction of the initial stage; A, change of weight, B, change of interplanar spacings of FeS.

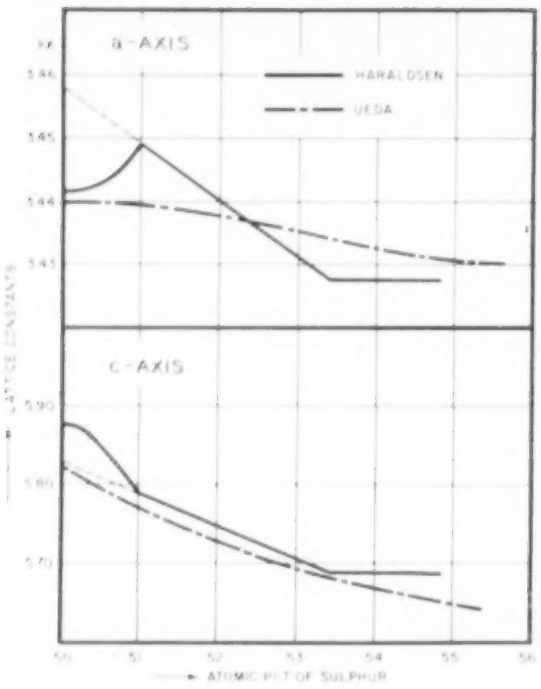


Fig. 3—The relation between the lattice constants and the atomic percentage of sulfur in $FeS_{1+x}$.

The lattice constants $a$ and $c$ of initial and oxidized ferrous sulfide are calculated from a set of two lines (103) and (202), using the well known equation for the hexagonal lattice

$$d_{hkl} = a/\left\{\frac{3}{4}(h^2 + hk + k^2) + (la/c)^2\right\}^{1/2} \quad [2]$$

where $d_{hkl}$ is the interplanar spacing of $(hkl)$. The results are shown in the first column of each specimen of Table III. The relation between lattice constants and the composition was determined by Haraldsen[3] and Ueda,[4] but there is a slight disagreement, particularly for $a$, between their results, as shown in Fig. 3. The second and the third column of each specimen in Table III give the atomic percentage of sulfur estimated by using these two results, respectively. Of the two results, Haraldsen's seems to be more reliable, since the agreements between the atomic percentage of sulfur calculated from $a$ and that from $c$ are better. Particularly, 51.4 pct of the initial specimen shows a good agreement with 51.7 pct obtained by the chemical analysis. From Table III, the increases of atomic percentage of sulfur are estimated to be $0.9 \pm 0.3$ pct and $1.4 \pm 0.3$ pct for 500° and 600°C, respectively. If Eq. 1 is assumed, the increase of atomic percentage of sulfur $\Delta S_{at}$ is given by

$$\Delta S_{at} = 100 \times \left\{\frac{1+\alpha}{2+\alpha-x} - \frac{1+\alpha}{2+\alpha}\right\} \quad [3]$$

and the increase in weight percentage $\Delta W_{wt}$ is

$$\Delta W_{wt} = 100 \times \frac{21.33\,x}{M_{FeS_{1+x}}} \quad [4]$$

where $M_{FeS_{1+x}}$ represents the molecular weight of $FeS_{1+x}$. From Eqs. 3 and 4, eliminating $x$, we obtain

$$\Delta W_{wt} = 100 \times \frac{21.33\,(2+\alpha)^2}{M_{FeS_{1+x}}} \left\{\frac{\Delta S}{\Delta S(2+\alpha) + 100(1+\alpha)}\right\}. \quad [5]$$

Using Eq. 5, and regarding the initial composition as 51.4 pct S, or $\alpha = 0.058$, the increases of weight $\Delta W_{wt}$ to be expected are $0.8 \pm 0.3$ and $1.3 \pm 0.3$ wt pct for 500° and 600°C, respectively, as far as Eq. 1 is assumed. The experimental values are 1.5 pct for both cases, and the agreement is good, particularly for the case of 600°C. The minor discrepancy at 500°C is probably due to the formation of the small amount of sulfate.

From these experimental results the mechanism of the initial reaction is conceived to be as follows. First, iron ions combine with oxygen, forming oxides on the surface. This stage is continued by migration of iron ions from interior of FeS crystal, and no evolution of $SO_2$ occurs, resulting in the deficiency of iron ions in FeS and an increase of weight. When the deficiency of iron reaches a limiting value, the production of $SO_2$ starts and weight begins to decrease.

## On the Rate of Oxidation

The rate-determining factor in this reaction is diffusion of gas, because the energy of activation is very small, as noted in the previous section. Schwab and Philinis[5] have considered that the observed rate is controlled by diffusion of oxygen through the pores of the product layer formed on the surface of each particle. The diffusion coefficient thus determined from their observations, however, was of the order of $10^{-7}$ $cm^2$ $sec^{-1}$, which was far less than the

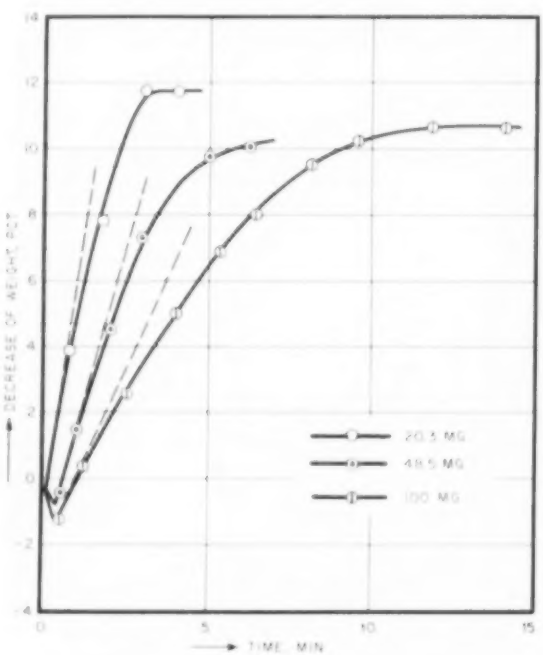


Fig. 4—Dependence of the rate of oxidation upon the amount of sample. The broken lines indicate the relative rate of oxidation cited in Table IV.

theoretical value of about $10^{-1}$ cm$^2$ sec$^{-1}$, indicating, according to their opinion, that the porosity of the product was very low.

This interpretation is rather open to question, however, because the rate may be determined not only by the diffusion in a particle, but also by that in the bed of the whole sample. The latter controlling factor was neglected by these authors.

If an attempt is made to determine the diffusion coefficient from the present observation in a way similar to Schwab's calculation, $D$ is found to be about $4 \times 10^{-4}$ cm$^2$ sec$^{-1}$, which is also a very small value. In this calculation, the fraction $\alpha$ of the pore or opening in the product was considered as

$$\alpha = 1 - \frac{V_{Fe_2O_3}}{V_{FeS}} \qquad [6]$$

where $V_{FeS}$ and $V_{Fe_2O_3}$ are the equivalent volumes of FeS and $Fe_2O_3$, respectively. Since sulfate was not formed in the present case, $\alpha$ may not be lowered very much.

An experiment was carried out to decide whether or not the diffusion in a particle is the only factor in the rate-determination; namely, the rate of oxidation was measured by using various amounts of the samples. If the diffusion in a particle is the rate-determining factor, the rate should be independent of the amount of sample. If, on the other hand, rate is controlled by the diffusion in the bed of sample, it may vary with the amount of sample. The results are shown in Fig. 4. They indicate that the rate depends on the amount of sample. Thus the observation supports the latter case of the two considerations mentioned above.

The shape of sample bed in the bucket was approximately a disk-type, with diameter from several millimeters to about 1 cm and a few millimeters thickness. The diffusion will occur in the direction normal to the upper surface of the disk, so the molar rate of oxidation should be proportional to the area of upper surface, or the area of extent of the sample bed in the bucket, as far as the diffusion in the bed is the rate-determining factor.

The second and third columns of Table IV record, respectively, relative and molar rate of oxidation determined from the slope of curves in Fig. 4, and $A$ in the fourth column is the area of extent of sample bed in the bucket. As shown in the fifth column of this table, the molar rate of oxidation per unit of $A$ is almost constant. This is a valid evidence for the view that the diffusion in the bed contributes predominantly to the rate of oxidation.

As a simple approximation, the rate of reaction $v$ controlled by the diffusion in the bed is

$$v = \alpha AD \frac{\Delta C}{\Delta L} \qquad [7]$$

where $\alpha$ is the fraction of the effective area in the apparent area $A$, $\Delta C/\Delta L$ is the concentration gradient of gas in the diffusion layer of the thickness $\Delta L$, and $D$ is the diffusion coefficient. Regarding $\Delta C$ as the concentration of oxygen in the air of 1 atm pressure and at the temperature of the reaction, it is about $2 \times 10^{-6}$ mol per cm$^3$. There is an uncertainty in $\Delta L$, which will vary from zero to the thickness of sample bed as the reaction proceeds. As a rough estimation, however, it can be assumed to be $10^{-1}$ to $10^{-2}$ cm. The $\alpha$ may be 1/2 to 1/5. If $D$ is of the order of $10^{-1}$ cm$^2$ sec$^{-1}$, the rate of reaction per unit area should then be $10^{-6}$ to $10^{-5}$ mol cm$^{-2}$ sec$^{-1}$. Although this is a very rough estimation, the order of magnitude is thus in good agreement with the experimental value given in the final column of Table IV.

It depends, of course, on complicated factors such as the particle size, the amount of the sample, and the shape of the bed, etc., whether the rate of reaction is predominantly controlled by the diffusion in the bed or by that in a particle. It is of interest, however, that the diffusion in the bed contributes even in such a system containing such small amounts of material.

## Summary

The rate and mechanism of oxidation of ferrous sulfide were studied by means of a spring balance and X-ray diffraction analysis over the temperature range from 500° to 700°C. The conclusions reached are as follows: 1) Complete oxidation, in which only the formation of oxides proceeds, begins at 600°C. 2) At the initial stage of oxidation, a slight increase of weight is found. It was observed by X-ray that the deficiency of iron ion in FeS occurs in this stage. This is interpreted as follows: Iron ion reacts with oxygen by migrating from the interior of the FeS crystal to the surface, without evolution of $SO_2$, until the deficiency of iron attains a limiting value. 3) Above 600°C the rate of oxidation does not change with temperature, so the rate-determining factor is diffusion of gas. Since the observed rate of oxidation depends upon the quantity of the sample, the diffusion in the bed of sample may determine the rate.

Table IV. Relative and Molar Rate of Oxidation for Various Amounts of Samples at 700°C

| Amount of Sample, Mg | $df/dt$, Sec$^{-1}$ * | $dm/dt$, Mol Sec$^{-1}$ ** | $A$, Cm$^2$ | $\frac{1}{A}\frac{dm}{dt}$, Mol Sec$^{-1}$ Cm$^{-2}$ |
|---|---|---|---|---|
| 20.3 | $0.76 \times 10^{-2}$ | $1.8 \times 10^{-6}$ | 0.35 | $0.51 \times 10^{-5}$ |
| 48.5 | $0.49 \times 10^{-2}$ | $2.7 \times 10^{-6}$ | 0.50 | $0.54 \times 10^{-5}$ |
| 100 | $0.30 \times 10^{-2}$ | $3.4 \times 10^{-6}$ | 0.70 | $0.49 \times 10^{-5}$ |

* $f$ is the fraction of oxidized ferrous sulfide.
** $m$ is the moles of oxidized ferrous sulfide.

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**Discussion** of this paper sent (2 copies) to AIME by Apr. 1, 1957 will appear in AIME *Transactions* Vol. 209, and in JOURNAL OF METALS, October 1957.

# Use of Oxygen At Abbey Melting Shop, Steel Co. of Wales Ltd.

A water-cooled probe, introduced through the roof of an open hearth furnace for jetting oxygen into the steel, is described. Effects on carbon elimination and other operating data are given. A system of slag control for sulfur elimination, involving control of oxygen utilization, lime addition and bath carbon, is described.

by A. J. Kesterton

MORE than 90 pct of the total tonnage of ingots made at Abbey Melting Shop is for steel sheet to specifications ranging between 0.055 and 0.07 pct maximum carbon. Since the rate of carbon elimination in the open hearth declines considerably as the carbon approaches these low levels, the use of oxygen for this purpose constituted a profitable field for development, particularly to increase ingot output.

## Development

Several experiments were carried out initially in which water-cooled guns, originally of all-steel construction and later with fabricated copper heads, were introduced manually through a hole in the back wall of one of the 225 net ton furnaces, the oxygen being delivered about 4 to 6 in. above the slag. Successful blows were obtained, but manipulation of the gun was cumbersome.

Application through the charging doors by means of bare steel lances was considered but rejected, because much of the scrap was light, necessitating a large number of buggies per charge, and consequent long occupation of the track in front of the furnaces by buggies.

To construct an efficient mechanism it was decided to experiment with the introduction of the gun through a hole in the roof. This was eventually successfully achieved, with the following advantages.

1) The actual mechanism required is simplified, and can easily be remotely controlled.

2) No large steel framework projects out over the back platform, possibly interfering with tapping, and there is no danger of damage by casting cranes.

3) The railway track in front of the furnaces is left entirely clear for the passage of scrap buggies, eliminating interference with adjacent furnaces charging.

4) As compared with the use of bare steel lances, a water-cooled jet eliminates the manpower required to handle, control and change the bare pipes.

5) A jet through the roof can be situated over the center of the bath so that, whatever the direc-

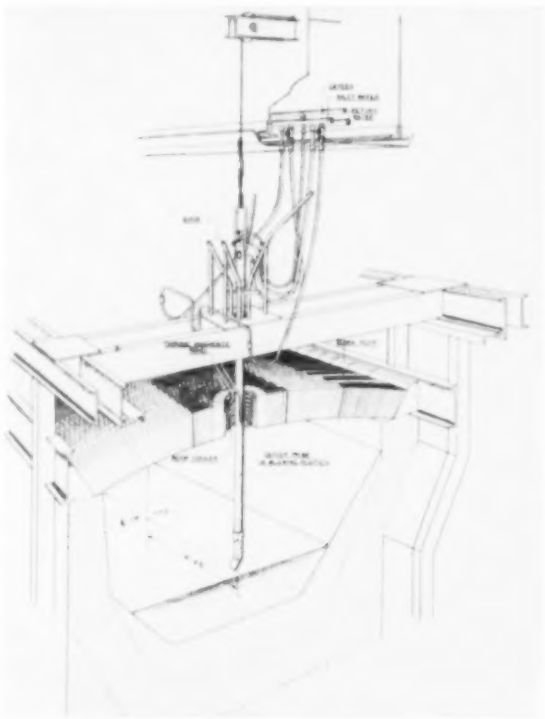

Fig. 1—Diagram of apparatus used in experiments.

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tion of flow in the furnace, there is a full half bath length available in which to burn the combustible gases arising from the oxygen reaction. If entry of a water-cooled gun is made through the back wall, this cannot generally be done over the taphole, and must be offset toward one end of the furnace, making combustion of the reaction gas less efficient from one direction.

## Description of the Equipment

Operation is simply effected by means of an electric winch situated inside the main gantry girder above the furnace, which moves a wire rope passing over a series of pulleys to the oxygen gun, see Fig. 1. The hoisting and lowering of the oxygen gun are carried out by remote control push button switches situated on the furnace instrument panel.

Attached to the gun near the top are two diametrically opposed steel pins which, as the gun is lowered, move into position between guides fixed to the top steel binders of the furnace. The guides consist simply of 2 in. diam steel tubes which are bent outwards to form a four-pronged splay to receive the pins. Two of the steel tubes are also lengthened and splayed in the direction at right angles, to act as a guide for and to gather together the hose pipes carrying the inlet and outlet water, and oxygen.

The steel pins, when located in the guides, prevent the oxygen gun twisting in any direction, and thus ensure that the gun fires longitudinally along the furnace, and is not directed toward front or back lining. The position of the pins on the gun is also such that should the wire rope break, they will strike the top binders, and prevent the end of the gun, which is water-cooled, dipping below the surface of the liquid steel.

Table I. Heat Losses Due to Water Cooling

| Cooling Unit | Water Flow, Imperial Gpm | Temperature Rise, °F | Heat Loss, BTU per Min |
|---|---|---|---|
| Gun retracted into roof | 106 | 1.5 | 1,590 |
| Gun lowered | 106 | 9.5 | 10,085 |
| Roof cooler | 48.7 | 5.0 | 2,435 |

The gun passes into the furnace through a hole in the roof, in which is fitted a water-cooled steel pipe spiral. This enters the 15 in. thick roof to a depth of about 9 in., the water being delivered to the lowest spiral, and then circulates upward around the spiral at high velocity, before being removed at the top. This design of roof cooler is efficient, in that it prevents excessive wear of the roof due to the flame passing through the hole, and it has a long life.

Surrounding the water-cooled spiral is one ring of chrome magnesite bricks, and in the longitudinal direction in which the oxygen blows, there is a further panel of chrome magnesite 5 ft 6 in. long by 4 ft 6 in. wide, which enhances roof life in this area. It is more resistant to any splashes which may reach the roof, and to the effects of flame arising at the reaction zone, the latter particularly if the oxygen is blown at high carbon. The present design of the oxygen gun is the result of trial and error development.

The gun, which is 4½ in. OD, is held vertically, and freely suspended from the wire hoisting rope. The lower end is cranked away from the vertical at an angle of 25°, the function of which is to ensure that the reaction zone is not immediately below the gun. This greatly reduces the quantity of slag and metal splashing back around the orifice, which is found to lengthen the life of the gun appreciably. The amount of crank in the end is such that the gun will pass freely through water-cooled annulus in the roof.

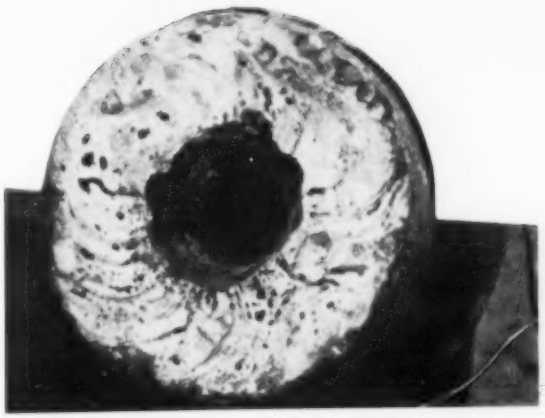

Fig. 2—Gun No. 13 after 75 blows, illustrating type of chamfering leading to failure.

The lower part of the gun for a length of about 16 in., the central 1 in. bore oxygen tube throughout its length, and the end plate are fabricated in copper, using copper welding.

Surrounding the oxygen tube is a cluster of three symmetrically situated ¾ in. bore steel pipes conveying cooling water to the end plate. These pipes pass through a diaphragm plate at the top of the gun and receive their water from a single large bore inlet pipe above the diaphragm. The return water is taken off below the diaphragm plate.

The average life of this design of gun is approximately 60 oxygen blows of about 40 to 50 min duration each. The record run without repairs is 236 blows. Failure usually occurs due to chamfering of the orifice at the point of exit of the oxygen, with eventual cutting through the tube and end plate into the water jacket. Repairs are, however, effected by copper welding quite cheaply, and the gun will run again for as many lives. This repair process has been repeated many times on some guns. As an example, one gun may be quoted which has had five spells of operation involving four repairs, the total lives to date being 298. Fig. 2 shows the type of chamfering leading to failure.

The water cooled gun and roof spiral subtract little heat from the furnace. See Table I.

## Operation

Operation by the first helper is extremely simple, lowering and withdrawal of the gun being effected by push button control from the instrument panel. The oxygen supply is controlled by the operation of a single valve from the same position.

The oxygen is produced at the present time by evaporators at a pressure of approximately 250 lb per sq in., which is reduced to 175 lb per sq in. to maintain steady conditions in the main.

However, in the near future, the oxygen will be supplied directly into a main running the full length of the valve house from a generating plant on the Works boundary operated by the British Oxygen

Table II. Rimming Steel, 0.07 Pct C Maximum

| Type of Heat | No. of Samples | CaO, Pct | $SiO_2$, Pct | $P_2O_5$, Pct | MnO, Pct | MgO, Pct | $Al_2O_3$, Pct | S, Pct | FeO, Pct | $Fe_2O_3$, Pct | Fe, Pct |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Oxygen | 51 | 42.92 | 6.22 | 4.65 | 4.38 | 6.24 | 3.14 | 0.23 | 19.71 | 9.81 | 22.11 |
| Non-Oxygen | 72 | 41.66 | 7.89 | 4.47 | 4.47 | 6.19 | 2.74 | 0.21 | 22.28 | 10.56 | 24.60 |
| Probability | | 0.061 | 0.32 | 0.27 | 0.71 | 0.88 | 0.061 | 0.075 | <0.001 | 0.13 | <0.001 |

Co. Ltd. Steady pressures up to 250 lb per sq in. may then be obtained if required.

On the instrument panel of each furnace there is an oxygen pressure gage measuring the pressure before the flow control valve, and a combined oxygen flow rate indicator and total flow integrator. The integrator has been arranged so that it can be reset to zero before each oxygen blow, in order that

Table III. Comparative Fettling Times, Rimming Steel, 0.07 Pct C Maximum

| Type of Heat | No. of Samples | Fettling Time, Min |
|---|---|---|
| Oxygen | 101 | 55.6 |
| Non-Oxygen | 101 | 78.4 |
| Probability | | 0.003 |

the number of cu ft of oxygen consumed can be read directly at any time during the process.

The use of oxygen is normally begun as soon as the bath is clear melted or virtually so, at any point below approximately 0.45 pct C. Above this level of carbon the reaction may become violent in the bath, producing a good deal of flame which, even with the oil completely off the furnace, may nevertheless locally raise the roof temperature high enough to melt the silica bricks. Also, the fume issuing from the stack is heavy. Occasionally, however, the oxygen may be used for a few minutes at a reduced rate of flow, at higher carbon levels than this, for the purpose of clearing up the final stages of the melt or promoting action in a sluggish bath.

However, below 0.35 to 0.40 pct C, the whole process in the Abbey open hearth furnaces is entirely manageable, and the discharge of fume from the chimney is usually less than that following the normal introduction of hot metal. Fume formation rapidly tails off, and below 0.20 pct C is slight.

Splash varies mainly with the height of the jet orifice above the bath, increasing as this distance is increased above an optimum figure of about 4 in. If the jet is lifted 9 or 10 in. above the bath a good deal of splash occurs in droplet form, which can and does reach the roof.

The oxygen flow rates used, assuming a straightforward refining condition, in which the sulfur is not a complicating problem (the main consideration is rapid carbon removal), are adjusted to the highest level which the bath will accept. This may mean, for instance, that if the oxygen is commenced at relatively high carbon, such as 0.45 pct, the flow rate will be about 40,000 cu ft per hr, in order for the reaction and flame produced to be controlled to a safe level. As the carbon drops, however, the oxygen flow rate will be increased to about 60,000 cu ft per hr.

It has been found that the delivery of a specific volume of oxygen, proportional to the carbon content at the commencement of the blow, will reduce the carbon to that required to meet the 0.07 pct maximum specification in the pit samples. Operation therefore is as follows.

A sample is sent to the laboratory at the same time as the oxygen blow is commenced, the integrator on the oxygen recorder being reset to zero. When the carbon analysis is available, the quantity of oxygen required is calculated, and as soon as this volume has been delivered, as read off directly from the integrator, the furnace is tapped without further reference to carbon analysis.

During the oxygen blow, the fuel oil input can normally be considerably reduced. It may vary from zero up to about two thirds of that required for normal operation, depending on bath conditions, the amount required being a matter of judgment. During the oxygen blow, bath temperatures are taken

Table IV. Comparative Sulfur Analyses (Pit Samples); Rimming Steel, 0.07 Pct C Maximum

| Type of Heat | No. of Samples | Pct S, Pit Analyses |
|---|---|---|
| Oxygen | 101 | 0.031 |
| Non-Oxygen | 101 | 0.031 |
| Probability | | 0.575 |

and serve as a guide to the oil flow required to supplement the heating effect of oxygen. However, with very little oil being used, the bath temperature may easily be increased from between 2805° and 2815°F to between 2885° and 2905°F during an oxygen blow from 0.35 to 0.40 pct C.

While the oil input is normally reduced during the oxygen blow, the air/fuel ratio is appreciably increased, in order for the CO generated at the reaction zone to receive sufficient air for burning as a supplementary fuel.

An experiment has been attempted to achieve the most efficient transfer of the heat from this supplementary fuel to the bath. It was thought that this could be done by providing a long path for the flame over the bath.

Instead of a single retractable gun placed over the center of the hearth, two guns were provided, enter-

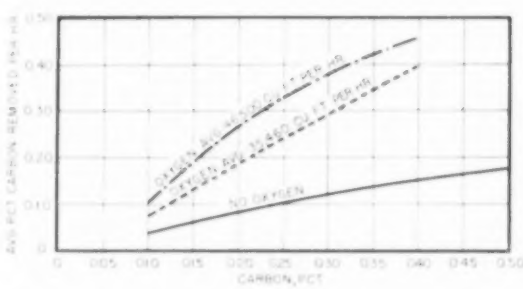


Fig. 3—Average rate percentage of carbon drop per hr for oxygen blown and normal heats.

ing the roof close to the ends of the bath. Oxygen was supplied alternately through these two guns, on the same end of the furnace as that through which the fuel and air were entering. The oxygen flow to each end was controlled by electrically operated valves connected to the automatic reversal system, so that reversal of the oxygen flow was also automatic. Therefore, approximately 80 pct of the bath length was always provided for combustion and heat transfer from the reaction gas.

However, no significant improvement in oil consumption was achieved, compared with the single central gun, indicating that a half bath length for combustion of the reaction gas is adequate. Since double the installation and gun maintenance costs were involved, all furnaces have now been converted to single central guns.

## Rates of Carbon Elimination

There is a considerable increase in the rate of elimination of carbon. The effect on the furnaces at Abbey is conveniently summarized in the graphs shown in Fig. 3. These show the average rate of carbon elimination in pct per hr from various carbon levels, calculated over the range from the bath carbon analysis at which the oxygen is commenced, to the bath analysis prior to tapping, which is approximately 0.06 to 0.07 pct for normal heats, and 0.05 to 0.06 pct for oxygen blown heats.

Three curves are shown, indicating the carbon elimination rates for heats blown at average rates of 35,460 cu ft O per hr, 46,500 cu ft O per hr, and non-oxygen heats, respectively.

Table V. Low Sulfur Steel Specifications; Maximum Sulfur, 0.027 Pct; 225 Net Ton Heats

| S at Melt, Pct | C at Melt, Pct | Add Hot Metal Long Tons | Feed Lime Boxes | Oxygen,* Flow Rates At 36,000 Cu Ft per Hr | Oxygen,* Flow Rates At 60,000 Cu Ft per Hr |
|---|---|---|---|---|---|
| 0.05 | 0.50 | 6 | 5/6 | | |
| 0.05 | 0.45 | 9 | 5/6 | | |
| 0.05 | 0.40 | 12 | 6 | 27,000 cu ft | Remainder |
| 0.05 | 0.35 | 14 | 6 | | |
| 0.05 | 0.30 | 15 | 6 | | |
| 0.05 | <0.30 | Divert | | | |
| 0.045 | 0.45 | 5 | 5 | | |
| 0.045 | 0.40 | 8 | 5 | | |
| 0.045 | 0.35 | 11 | 5/6 | 24,000 cu ft | Remainder |
| 0.045 | 0.30 | 13 | 6 | | |
| 0.045 | 0.25 | 15 | 6 | | |
| 0.045 | <0.25 | Divert | | | |
| 0.040 | 0.45 | Nil | 4/5 | | |
| 0.040 | 0.40 | 5 | 4/5 | | |
| 0.040 | 0.35 | 8 | 4/5 | 21,000 cu ft | Remainder |
| 0.040 | 0.30 | 10 | 5 | | |
| 0.040 | 0.25 | 12 | 5 | | |
| 0.040 | 0.20 | 15 | 5 | | |
| 0.040 | <0.20 | Divert | | | |
| 0.035 | 0.45 | Nil | 4 | | |
| 0.035 | 0.40 | Nil | 4 | | |
| 0.035 | 0.35 | 5 | 4 | | |
| 0.035 | 0.30 | 8 | 4 | 18,000 cu ft | Remainder |
| 0.035 | 0.25 | 10 | 4/5 | | |
| 0.035 | 0.20 | 12 | 5 | | |
| 0.035 | 0.15 | 15 | 5 | | |
| 0.030 | 0.45 | Nil | 3 | | |
| 0.030 | 0.40 | Nil | 3 | | |
| 0.030 | 0.35 | Nil | 3 | | |
| 0.030 | 0.30 | Nil | 3 | 15,000 cu ft | Remainder |
| 0.030 | 0.25 | 6 | 3 | | |
| 0.030 | 0.20 | 9 | 3 | | |
| 0.030 | 0.15 | 12 | 4 | | |

* Total required cubic feet are as required by carbon after hot metal addition, if made. see Table VI.

The beneficial effects of using oxygen are immediately apparent. At the higher level of oxygen flow rate, i.e., 46,500 cu ft per hr, the carbon elimination rate is increased approximately threefold

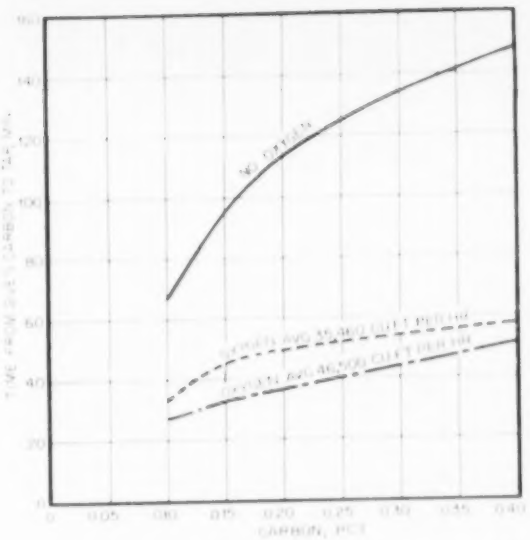


Fig. 4—Time from given percentage of carbon to tap for oxygen blown and normal heats.

throughout the whole range, as compared with non-oxygen heats.

In Fig. 4 graphs are given, showing the refining time required from a given carbon to tapping for both non-oxygen and oxygen blown heats, the latter at the two blowing rates. As an example, from 0.35 pct C, the use of oxygen at the higher rate reduces the time to tap from 140 to 48 min. From 0.15 pct C the corresponding reduction is from 96 to 33 min.

In Fig. 5 the time saved for oxygen-blown as compared with non-oxygen heats is graphed over the full range of carbon levels, and for the higher oxygen flow rate varies from 40 min saved at 0.1 pct C to 95 min at 0.40 pct C.

Fig. 6 shows the specific consumption of oxygen in cu ft per 0.01 pct C per long ton of steel, plotted against the percentage of carbon at the start of the blow. From this it is quite clear that there is a rapid reduction in the efficiency of the use of oxygen as the percentage of carbon at the start of blow declines. The reduction in efficiency is at a spectacular rate below 0.15 pct C.

This fact, together with the increase in time saved the higher the percentage of carbon at which oxygen blowing is commenced, suggests that the higher the carbon at which blowing is commenced the greater is the net gain from all points of view. This is counterbalanced by the physical fact that above 0.45 pct C the reaction is too violent, and accordingly approximately 0.40 pct C has been selected as the best compromise at which to commence oxygen blowing.

## Tapping Slag Analysis

In Table II a comparison is made between the average tapping slag analyses for 51 oxygen blown heats and 72 non-oxygen heats. The slag analyses are routine determinations from heats of the same grade of steel, taken at random from the furnaces during the same period of time. Two furnaces were operated with oxygen, and six on ore heats.

The probability that the difference between the various analyses is not significant is given below each slag constituent. It is clear that the differences

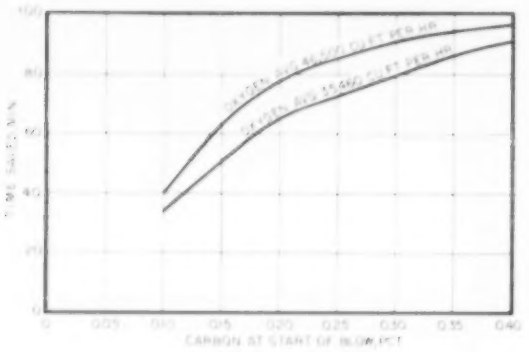

Fig. 5—Time saved from given percentage of carbon to tap for oxygen blown compared with normal heats.

shown for CaO, Fe, FeO, and $Al_2O_3$ have only one chance in 1,000 of being random.

There is therefore a useful significant difference in the iron oxide concentration favorable to the oxygen blown heats. Expressed as iron, this constituent is about 2.5 pct less for the oxygen heats.

Also significantly different, and probably connected with the slag percentage of iron being lower, the percentage of CaO is higher by 1.92 pct in the oxygen heats.

The oxygen heats therefore work with a slag lower in iron oxide, but higher in CaO, to produce the same carbon specification in the steel. Accordingly, if other things are equal, the iron loss in the slag will be reduced, and the steel yield slightly increased.

## Fettling

There is a good indication of a reduction in fettling time resulting from the use of oxygen.

This probably arises from the shorter refining times during which the hearth refractories are exposed to the most severe conditions of temperature and high iron oxide concentration in the slag. Also the oxygen jet generates heat directly into the metal bath itself which, together with the lower fuel inputs, appears to give lower slag temperatures. Accordingly, reduction in fettling time may be reasonably expected.

Table III compares the average fettling time for 101 oxygen and 101 non-oxygen heats. The selected samples are successive heats of the same grade of steel, i.e., a maximum of 0.07 pct C rimming steel, made during the same period. The oxygen heats are made on two furnaces fitted up to use oxygen, and the non-oxygen heats are made on the remaining six furnaces. In the latter case an approximately equal number of heats was selected from each furnace.

## Sulfur

Table IV shows the average percentage of sulfur of the pit samples for 101 oxygen heats compared with the same number of non-oxygen heats. The selected samples are the same heats used in Table III for the comparison of fettling times.

There is no significant difference between the oxygen and non-oxygen heats. However, it is interesting that the same sulfur elimination is secured in oxygen blown heats, even though the refining time is cut to approximately one third.

**Roof Life**—If properly applied as described in the text, the use of oxygen does not cause splashes which affect the roof or linings of the furnace.

**Sulfur as a Complicating Factor**—Abbey Melting Shop operates on relatively high sulfur fuel oil, i.e., 1.6 pct S. Much of the scrap is light, which lengthens charging times and increases sulfur pick-up from the fuel. There is 50 to 55 pct of high phosphorus (0.70 pct) and low sulfur hot metal (0.02 to 0.025 pct). Under these circumstances sulfur becomes a complicating factor for steel specifications less than 0.03 pct S.

The extent of the problem of refining sulfur depends upon the percentage of sulfur at melt in relation to the tapping analysis required. If the percentage of sulfur at melt is close to the final specification, the carbon may be confidently removed at high speed by the use of oxygen. On the other hand, if the final specification required is low and difficult to attain, e.g., less than 0.027 pct, and the percentage of sulfur at melt is relatively high, e.g., 0.04 to 0.045 pct or more, then experience dictates that the bath be refined primarily in relation to sulfur removal, and the elimination of carbon subordinated to this end.

It is thought, however, that this need not necessarily curtail the use of oxygen for carbon removal during refining, and the following analysis of the problem of refining sulfur is put forward for discussion, in which the use of oxygen is proposed as an essential tool.

The percentage of sulfur at melt will vary due to the influence of the variable operating factors up to

Table VI. Oxygen Volumes for Carbon Elimination to Obtain 0.05 Pct C at Tapping, Assumed Steel Weight, 235 Net Tons

| Carbon, Pct | Oxygen, Cu Ft |
|---|---|
| 0.45 | 42,000 |
| 0.40 | 38,500 |
| 0.35 | 35,000 |
| 0.30 | 32,000 |
| 0.25 | 29,000 |
| 0.20 | 26,250 |
| 0.18 | 25,500 |
| 0.16 | 24,800 |
| 0.14 | 24,000 |
| 0.12 | 23,000 |
| 0.10 | 22,000 |

that time, including the variable pickup of sulfur from the fuel, the variable condition of the scrap in respect to entrained silica and sulfur-bearing constituents, variations in the hot metal analysis for silicon, phosphorus, and sulfur, variable analyses and quantities of flush slag, and variable carbon percentages in melt outs.

The extent of the refining problem in relation to sulfur when aiming for a given specification is not known, therefore, until melt-out or close to it.

It is further suggested that the melt percentage of sulfur is self adjusting to the above factors, and that this figure is therefore proportional to the total sulfur in the system at melt-out and the sulfur carrying capacity of the slag. If the melt percentage of sulfur is higher than the steel specification, the capacity of the slag to remove sulfur must be increased during the refining period, and it is suggested that the amount of alteration which has to be effected during the refining period to the slag bulk and composition can be equated fairly closely to the difference between melt percentage of sulfur and final steel percentage of sulfur specification.

This therefore determines, in the first place, the amount of lime which it is necessary to put into

solution in the slag during the refining period, since this is the only convenient addition which can be made deliberately to the slag to increase its total sulfur-carrying capacity.

Whatever lime is used must first be put into fluid solution in the slag, following which there must be adequate slag/metal interfacial exchange or agitation to ensure that the sulfur is transferred from metal to slag.

The operation of dissolving the lime is one requiring the absorption of heat to raise it to slag temperature, together with a thorough mixing with the slag to facilitate solution. It is thought that, initially, the best procedure is not an all-out attack on the carbon with oxygen, but that the lime should be helped into solution by a combination of a reasonable time exposure to the flame and a carbon boil vigorous enough to ensure that all the lime is continuously surrounded by moving slag. It is proposed that the provision of these requirements, without excessive loss of carbon, should be secured by blowing with oxygen at a moderate rate initially. For instance, in the Abbey furnaces a rate of 36,000 cu ft per hr is selected at this stage. The length of time during which the moderate blowing rate is maintained to get the lime into solution is proportional to the lime addition made, and hence to the melt percentage of sulfur. It should be noted that a quantity of carbon will be blown out at this stage which is proportional to the volume of oxygen used at this low rate, and hence also to the lime addition and melt percentage of sulfur.

Having dissolved the lime, a further period of vigorous agitation betwen slag and metal to provide an adequate amount of slag/metal interfacial exchange for completing the sulfur transfer must be provided. The slag being fluid and reactive at this stage, it is thought that the agitation may be provided rapidly. The proposal is to step up the oxygen delivery rate to 60,000 cu ft per hr, i.e., twice that used to put the lime into solution. In any case, the carbon will be getting lower, and a higher rate of blowing is required to maintain a comparable rate of agitation. For this stage also, therefore, an amount of carbon must be available which will provide the necessary quantity of agitation.

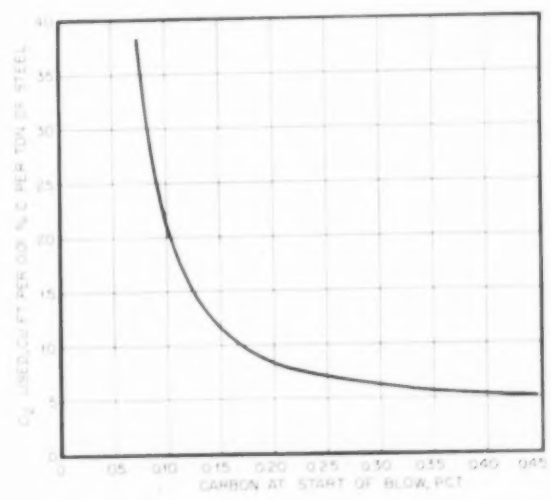


Fig. 6—Specific oxygen consumptions in cubic feet per 0.01 pct C per long ton steel for various percentages of carbon.

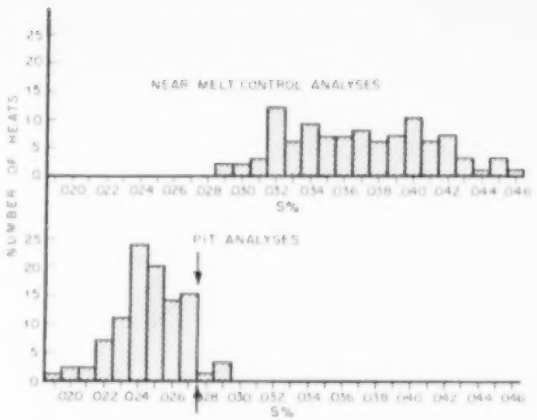


Fig. 7—Degree of success attained in sulfur slag control in 100 heats.

Therefore, there must be available at the melt an amount of carbon proportional to the melt percentage of sulfur, which will be sufficient to put the necessary lime into solution, and then adequately agitate the slag with the metal. If this carbon is not present at the melt, it should be added by hot metal addition. The quantities of hot metal proposed in Table V have been calculated from a statistical examination of data.

The proposed slag control method for sulfur removal during refining, using oxygen as an essential tool, is as follows.

1) Analyze a bath sample for carbon and sulfur as near to the clear melted stage as possible. Slightly before is preferable so that action based on the analyzed result can be taken immediately when the bath is melted.

2) Depending upon the sulfur analysis, two factors will be determined, i.e., the amount of lime to be fed, and the amount of carbon required to provide the necessary agitation. The greater the melt percentage of sulfur, the greater the lime required, and the higher the percentage of carbon necessary. If the carbon is below that required, a hot metal addition is made.

3) Depending on the lime addition, a specific proportion of the total oxygen volume required is blown at a reduced rate to get the lime into solution. After this, the remainder of the oxygen is blown at a high rate.

4) The total oxygen required is determined by the carbon analysis after hot metal addition (if any), at the moment the oxygen blow is started. The completion of the refining can then proceed without further reference to carbon or sulfur analysis, the furnace being tapped at the end of the oxygen blow.

5) Tables V and VI have been prepared laying down the hot metal addition, lime addition, oxygen blowing rates, and oxygen volumes required for specific carbon analyses.

The refining procedure is given in the Appendix. To date the proposals are in a tentative experimental stage subject to change in the light of experience, and are put forward at this juncture mainly for the purpose of indicating that it is recognized that the refining of sulfur is a complicating factor in the use of oxygen for decarburization, and also to draw critical comments, which it is hoped

will help to effect improvement. Results to date, however, indicate that the desired sulfur specification can be achieved successfully, using the proposed system.

Fig. 7 shows the degree of success attained in securing the desired sulfur specification of 0.027 pct maximum in 100 heats operated in this way. The number within specification is 96 pct. Hot metal additions were made, as part of the process, to 33 pct of the heats.

### Quality

Oxygen is now used for the manufacture of low carbon sheet steel, including the highest qualities of extra deep drawing, fully stabilized, etc., with no observable difference in quality as compared with previous practice.

In the case of steel for tinplate, the Tinplate Division reports that the oxygen blown coils pickle well, and have a lower than average Rb hardness figure for the hot rolled band. They also roll very well in the 5-stand cold mill, with fewer instances of delay. Performance at the temper mill is reported as excellent, with fewer coils needing to be rerolled for fluting, and the tempered Rockwell hardness figures are well grouped together in the center of the range.

### Appendix: Low Sulfur Steel Specifications—Maximum Sulfur, 0.027 Pct

**Refining Operations Using Oxygen**—1) Prior to 30 min before melt, the bath is worked in the usual way so far as additions of oxide, hot metal, etc., are concerned, but a melt carbon of about 0.50 pct is aimed for.

2) Approximately 30 min before melt, a sample is sent to the laboratory for analysis of carbon, sulfur, phosphorus, and manganese.

3) Approximately 15 min before melt, a sample is sent to the laboratory for carbon only.

4) While waiting for these samples, lime is fed to the bath, if the condition is suitable. This lime counts as part of the total addition required as calculated later from Table V.

5) When the bath is melted, the above two bath samples should be ready, i.e., one sample for sulfur, 30 min before melt, and two samples for carbon, one 30 min and one 15 min before melt. The percentage of carbon at melt can then be deduced from the two samples, and the percentage of sulfur at melt can be estimated by deducting 0.002 pct from the result of the first sample. For example, first sample, 30 min before melt, 0.45 pct C, 0.043 pct S; second sample, 15 min before melt, 0.40 pct C, no sulfur. Therefore, at melt 0.35 pct C and 0.041 pct S is estimated.

6) Table V is now read to deduce the action required. In the example given above, for 0.35 pct C and 0.041 pct S at melt, the additions required are eight tons of hot metal, and four or five boxes of lime.

7) The hot metal is added as quickly as possible, giving more of the lime with the hot metal, to make use of the boil from the latter.

8) Oxygen blowing is begun as soon as the bath subsides after hot metal addition, and a sample is sent to the laboratory for carbon analysis only, which determines the volume of oxygen to be used.

9) The remainder of lime required is added *as early as possible* in the oxygen blow.

10) Oxygen is blown at the reduced rate of 36,000 cu ft per hr for the period specified in Table V to put the lime into solution, and then increased to 60,000 cu ft per hr for the remainder of the oxygen required.

11) The furnace is tapped as soon as the oxygen volume specified in Table VI has been delivered, without sending further samples to the laboratory for carbon and sulfur analysis. However, a running-out sample is sent to the laboratory for carbon, sulfur, and manganese analyses.

12) Fluorspar is used with lime additions to assist rapid solution.

**Discussion** of this paper sent (2 copies) to AIME by Apr. 1, 1957 will appear in AIME Transactions Vol. 209, 1957 and in Journal of Metals, October 1957.

# Some Aspects of The Physical Chemistry of Hydrometallurgy

**Some of the principles of physical chemistry underlying hydrometallurgical processes are discussed and an attempt is made to evaluate the various thermodynamic and kinetic factors which influence their operation. Recent research in this field is reviewed and a number of specific leaching and precipitation reactions are discussed in detail. The application of kinetic measurements to the elucidation of reaction mechanisms is emphasized.**

by J. Halpern

PHYSICAL chemistry contributes to the understanding and efficient operation of hydrometallurgical processes in many ways, among them by providing quantitative answers to the following questions.

1) Can a given chemical reaction or process be made to operate and, if so, under what conditions?

2) What are the factors which determine the rate of the reaction and how can this rate be regulated in practice?

Thermodynamics attempts to answer the first of these questions and kinetics the second. The answer

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to the latter is intimately related to considerations of the mechanism by which the reaction takes place. An understanding of the mechanism is therefore of great importance. Sometimes a given reaction may occur by alternative mechanisms depending on conditions and on what catalysts, if any, are present.

A number of hydrometallurgical processes have been used for many years and the practices relating to their application are well established, although in most cases a detailed understanding of the physical chemical principles which govern their operation has not yet been achieved. Examples of common leaching processes are: the extraction of gold and silver by cyanidation; leaching of zinc oxide and copper oxide with acid; leaching of metallic copper with ammonia; leaching of alumina with caustic soda; and leaching of uranium with acid or carbonate. The common precipitation processes include: the cementation of copper from acid solution by iron; precipitation of gold and silver from cyanide solutions by zinc dust; precipitation of uranium and vanadium from either acid or carbonate solutions by neutralization; precipitation of insoluble sulfides; and many other chemical precipitation processes.

In recent years, interest in hydrometallurgical processes has been greatly intensified. Many of the newer developments in this field involve leaching and precipitation operations which are conducted at elevated temperatures and pressures.[1-3] The advantages which may result from operating under autoclave conditions are 1) greatly increased rates of reaction, 2) favorable displacement of thermodynamic equilibria, and 3) the possibility of using certain gaseous or highly volatile reagents such as $O_2$, $H_2$, and $NH_3$. Among the new processes which have resulted from the application of high temperature, high pressure techniques are: the direct leaching of nickel, copper, and cobalt sulfides with ammonia;[4-6] carbonate leaching of pitchblende ores;[7-9] leaching certain sulfide ores with water, all the necessary acid being generated *in situ* by autoxidation of the sulfides;[10-11] the precipitation of metallic nickel, cobalt, and copper from aqueous solutions of their salts by hydrogen reduction;[4,10-12] and precipitation of uranium and vanadium oxides from aqueous carbonate solution by hydrogen reduction.[8,13-14]

This paper is concerned in large measure with these newer developments in the field of hydrometallurgy, although recent advances relating to the physical chemistry of some of the older hydrometallurgical processes are also reviewed. Electrolytic processes which have been widely employed to recover metals from solutions, often in the purest obtainable forms, are excluded from the present discussion, as are some of the newer recovery techniques such as ion exchange and solvent extraction. Also excluded from consideration are the many important problems of physical chemistry connected with mineral dressing, a field very intimately associated with hydrometallurgy.

## Thermodynamic Considerations

Through the use of thermodynamics it is possible to define the equilibrium in a chemical system and to predict how the equilibrium will shift with changing conditions such as temperature, pressure, concentration, etc. In general an increase in the concentrations or partial pressures of the reactants will shift the equilibrium of a reaction in the forward direction, while an increase in the concentrations of the products will have the reverse effect. Where any of the reactants or products are gases or highly volatile substances, the role of pressure in influencing the thermodynamics becomes readily apparent. The principles and procedures involved in such thermodynamic calculations are well known, and several comprehensive tabulations of thermochemical data are available.[15-16]

The thermodynamic aspects of a number of systems of hydrometallurgical interest have been discussed in detail by Pourbaix,[17] and conveniently represented by him in the form of potential-*pH* diagrams. A typical diagram for the system, Cu-$H_2O$, defining the regions of stability of different forms of copper as functions of the potential and *pH*, is shown in Fig. 1. For details of construction of the diagram, the reader is referred to Pourbaix.[17] Following the convention used by him, the boundaries have been drawn so that the solution regions correspond to total concentrations of dissolved copper exceeding $10^{-6}$ mol per liter. Because of the logarithmic nature of the dependence of both *pH* and potential on the concentration, these boundaries are shifted only slightly if a different concentration limit is selected. On the same diagram the thermodynamics of the reduction of oxygen (at 1 atm) are represented, i.e.

$$O_2 + 4\,H^+ + 4e = 2\,H_2O \qquad [1]$$

and the oxidation of hydrogen (at 1 atm), i.e.

$$2\,H^+ + 2e = H_2 \qquad [2]$$

From the diagram the following thermodynamic information is readily apparent.

1) Throughout the *pH* range considered (0 to 14) metallic copper is thermodynamically stable in contact with an aqueous solution, in the absence of oxygen or other oxidizing agents; i.e., the potential for the oxidation of copper lies appreciably above that for the liberation of hydrogen.

2) Oxidation of the copper can be effected by gaseous oxygen at ordinary pressures, since the potential for the reduction of oxygen lies well above that for the oxidation of copper throughout the diagram. Several oxidation products are possible, corresponding to different reaction paths, which may be represented as follows:

$$Cu + \tfrac{1}{2}O_2 + HOH \rightarrow \begin{cases} Cu^{++} + 2\,OH^- & [3a] \\ CuO + HOH & [3b] \\ CuO_2^{--} + 2\,H^+ & [3c] \end{cases}$$

In the *pH* region between about 6 and 13, the insoluble oxides, $Cu_2O$ and CuO, are thermodynamically favored, while at lower and higher *pH* values, the copper can dissolve as $Cu^{++}$ and $CuO_2^{--}$, respectively.

3) All the oxidized forms are thermodynamically reducible to metallic copper by gaseous hydrogen at ordinary pressures, since the potential for the oxidation of hydrogen lies well below that for copper throughout the diagram.

The properties of the system can be profoundly influenced by the presence of a reagent which forms a complex ion of the metal in the solution. This is shown in Fig. 2, which depicts the potential-*pH* diagram for copper in contact with an aqueous solution containing 1 mol per liter total $NH_3$ (i.e., $NH_3 + NH_4^+$). The concentration of free $NH_3$ (the species

which complexes with $Cu^{++}$ is uniquely defined by the *pH* of the solution through the equilibrium

$$NH_4^+ + H^+ \rightleftharpoons NH_4^+ \quad [4]$$

At *pH* = 3 the concentration of free $NH_3$ is negligible, while above *pH* 9 it approaches the limiting value of 1 mol per liter.

The diagram defines the regions of stability of all the forms of copper, including the various cuprous and cupric ammine complexes. The solution regions correspond to conditions under which the total dissolved copper concentration (including simple and complex forms) can exceed $10^{-6}$ mol per liter. Among the notable effects arising from the presence of $NH_3$, evident from a comparison of Figs. 1 and 2, are:

1) The potential for the oxidation of metallic copper is generally lowered because of complex formation. Thus, from a thermodynamic standpoint, leaching is greatly facilitated, while reduction with hydrogen, although still feasible, becomes more difficult.

2) The region of insolubility of the oxide phases is greatly reduced. From the standpoint of leaching, this is a great advantage.

In addition to those given here, potential-*pH* diagrams have been published for the following metals: silver,[5,6] chromium,[7] zinc,[8] lead,[9] aluminum,[10] arsenic,[11] gold,[12] beryllium,[13] cadmium,[14] cobalt,[15] mercury,[16] selenium,[17] tin,[18] titanium,[19] and thallium.[20] In principle, such diagrams can also be constructed for systems involving minerals, such as oxides, sulfides, etc., in contact with aqueous solutions, and they should prove very helpful in understanding the thermodynamic problems connected with the leaching of these minerals. The applicability of potential-*pH* diagrams to other processes of hydrometallurgical interest, including chemical and reductive precipitation, electrolysis, and corrosion, is also readily apparent. Unfortunately the construction of such a diagram is often a laborious process, particularly for systems in which many compounds can be formed or in which such effects as complexing are taken into account. A further obstacle is the lack of reliable thermodynamic data for many systems, particularly in concentrated solutions and for the higher temperatures which are often of interest to the hydrometallurgist.

## Kinetic Considerations

In general, hydrometallurgical processes, particularly leaching reactions, are conducted under conditions in which the thermodynamics are very favorable (i.e., there is a large decrease in free energy associated with the desired process) and the limitations are therefore largely of a kinetic nature. In particular, where the thermodynamics of several alternative reactions do not differ greatly, kinetic factors may determine what products are formed. Because of these considerations, it appears increasingly important to understand the kinetic aspects of hydrometallurgical processes. However, up to the present only limited progress has been made in this direction.

Most hydrometallurgical processes, whether they relate to leaching or precipitation, involve heterogeneous reactions occurring at a solid-liquid interface, and in all but a few special cases, such reactions are rate-determining. Most of the heterogeneous reactions of interest in the present connection may be regarded as involving the following sequence of steps: 1) absorption of gaseous reactants, if any, by the solution; 2) transport of dissolved reactants from the main body of the solution to the solid-solution interface; 3) adsorption of reactants on the solid surface; 4) reaction on the surface; 5) desorption of the soluble products from the surface; and 6) transport of the desorbed products into the main body of the solution.

There appear to be only a few systems (one reported example involves the initial stages of ammonia leaching of a nickel sulfide concentrate[1]) in which step 1, the absorption of a gaseous reactant by the solution, is rate-limiting. In most cases, sufficiently fast gas absorption can be maintained* by using efficient agitators for the solution to remain saturated with the gas, and subsequent steps in the process become rate-determining. This is particularly true where 1) the partial pressure of the gas is high, 2) the pulp density is not excessive, and 3) the temperature is not too high (since the rates of most chemical reactions have a higher temperature coefficient than that of gas absorption).

* This becomes increasingly difficult with increasing size of the equipment used.

The transport of a dissolved reactant to the surface, step 2, or of a product away from the surface, step 6, is more often rate-determining. The essential features of systems to which this applies are depicted in Fig. 3, which shows how the solution concentration of the pertinent species varies as a function of distance from the surface. In each case, the rate of transport to or from the surface is determined by the diffusion of the species across a diffusion layer of thickness $\delta$, immediately adjacent to the surface at which it is consumed or formed. Within this layer, the concentration of the diffusing species can be represented to a first approximation as a linear function of the distance (corresponding to the broken line in Fig. 3), while on the solution side, its value is that for the bulk of the solution.

On the basis of this approximate model, the rate at which a dissolved substance diffuses to a solid surface (where it is consumed by reaction) is represented mathematically by Fick's law

$$\frac{dn}{dt} = \frac{DA}{\delta}(C - C_s) \quad [5]$$

where $n$ is the amount of diffusing material which moves across the diffusion layer in time, $t$; $D$ is the diffusion coefficient (usually of the order of $2 \times 10^{-5}$ sq cm per sec;[21] the value of $D$ increases only slightly with temperature); $A$ is the area of the diffusion layer (i.e., of the surface); $C_s$ is the concentration at the surface; and $C$ is the bulk solution concentration.

The diffusion rate reaches a limiting value given by

$$\frac{dn}{dt} = \frac{DA}{\delta}C \quad [6]$$

when the solution at the interface is completely depleted (by reaction) of the diffusing species, and hence $C_s = 0$. This represents an upper limit for the reaction rate.

Frequently the dependence of the rate on the concentration of a leaching reagent is of the form shown in Fig. 4. This figure depicts the rate of solution of copper in aqueous ammonia as a function of the oxygen partial pressure (or concentration).[22] The rate is initially determined by the diffusion of oxygen to the copper surface and, in accordance with Eq. 6, is directly proportional to the oxygen concentration (region *A*). However, as the oxygen concen-

tration (and consequently its diffusion rate) is increased, a point is reached where the leaching rate becomes determined by a chemical process which does not involve oxygen and hence is independent of the oxygen concentration. This corresponds to region *B*. Increased agitation increases the rate in region *A*, but does not affect the limiting rate, *B*.

A similar dependence of leaching rate on oxygen concentration has been observed for the cyanidation of silver.[7] However, in this system the oxygen-independent limiting rate corresponds to a condition in which the diffusion of cyanide, instead of oxygen, has become rate-determining.

For a given system, the thickness of the diffusion layer, $\delta$, depends primarily on the agitation. Its value normally ranges from about 0.05 cm in an unstirred system to about 0.001 cm in a vigorously agitated one.[8] Using these values it can be shown that the maximum attainable (diffusion-limited) rate of a heterogeneous reaction is of the order of $10^{-1}$ mol per sq cm per hr when the concentration, $C$, of the diffusing reactant is 1 mol per liter, or about $10^{-4}$ mol per sq cm per hr when one of the diffusing reactants is a gas such as oxygen or hydrogen whose concentration is unlikely to exceed $10^{-3}$ mol per liter except at very high pressures.

A number of criteria have been advanced to distinguish diffusion-controlled reactions from those which are chemically controlled. In contrast to the latter type, the rates of diffusion-controlled reactions are usually characterized by a marked dependence on agitation and by a low temperature coefficient, usually corresponding to an apparent activation energy between 1 and 4 kcal per mol.

Diffusion of a soluble product away from the reacting surface is less often rate-determining, although such cases are known.[9] The rate is represented by

$$\frac{dn}{dt} = \frac{DA}{\delta}(C_s - C) \qquad [7]$$

where the symbols have the same meaning as in Eq. 5. $C_s$, and hence the diffusion rate, can increase until the solubility of one of the products is exceeded, causing it to precipitate. For example, during the leaching of metallic copper, Eq. 3, slow diffusion may result in localized accumulation of $Cu^{++}$ and $OH^-$ in the vicinity of the metal-solution interface with the resulting precipitation of CuO even when the bulk solution concentrations favor the formation of soluble products. Once formed, the precipitated oxide redissolves slowly, and hence tends to passivate the copper surface.

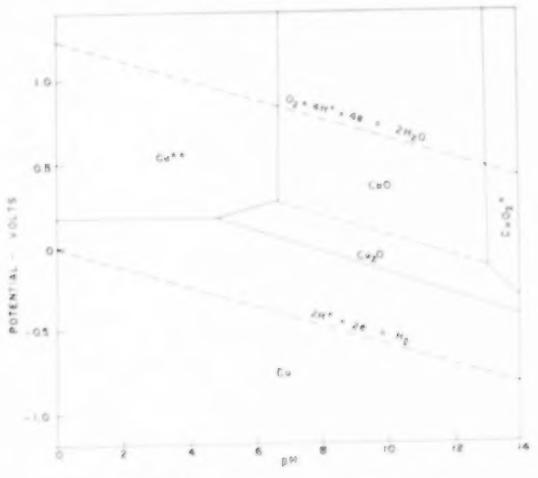


Fig. 1—Potential-*pH* diagram for the Cu-H$_2$O system.

Steps 3 through 5 (those involving adsorption, chemical reaction at the surface, and desorption) vary greatly from system to system and do not lend themselves to consideration in general terms. Among the complications which may arise in this connection is the formation of insoluble products (sometimes resulting from side reactions) which often accompanies leaching. As in the example discussed above, the leaching of copper, such products may tend to deposit on the surface of the reacting solid and form a passivating layer, through which the reactants and products must diffuse in order for the reaction to be sustained. Such diffusion, which is usually much slower than the liquid diffusion discussed earlier, even where the solid layer is fairly porous, may become rate-determining.

These and other features of the kinetics and mechanisms of hydrometallurgical reactions are best considered for individual systems, a number of which are discussed below.

## Leaching Reactions

**Cyanidation of Silver and Gold**—Both the thermodynamics and kinetics of the cyanidation of silver have been examined.[10] The principal reaction appears to be

$$2\,Ag + O_2 + 4\,CN^- + 2\,H_2O \rightarrow 2\,Ag(CN)_2^- + 2\,OH^- + H_2O_2 \qquad [8]$$

although it has been suggested[11] that at high $H_2O_2$ concentrations the following reaction may also contribute

$$2\,Ag + H_2O_2 + 4\,CN^- \rightarrow 2\,Ag(CN)_2^- + 2\,OH^- \qquad [9]$$

Under all conditions investigated, the diffusion of one of the reactants ($CN^-$ or $O_2$, depending on their relative concentrations) to the silver surface was found to be rate-limiting. The measured activation energy was 2 to 3 kcal per mol. Thus the chemical reaction appears to be very fast, and kinetic measurements fail to provide information about the chemical mechanism. It was established[12] that the adverse effect of lime on the cyanidation of silver is due to the formation of a protective deposit of insoluble calcium peroxide on the silver surface

$$H_2O_2 + Ca(OH)_2 \rightarrow CaO_2 + 2H_2O \qquad [10]$$

Similar considerations apply to the cyanidation of gold. Kudryk and Kellogg[13] have shown this to be an electrochemical process, and found the rate of reaction and the potential of the dissolving metal to be in agreement with the values predicted from anodic and cathodic polarization measurements. Their results suggest that the principal reaction is

$$4\,Au + 8\,CN^- + O_2 + 2\,H_2O \rightarrow 4\,Au(CN)_2^- + 4\,OH^- \qquad [11]$$

However, other investigators[14] have claimed the formation of $H_2O_2$ as an intermediate and confirmed its presence analytically. The reason for this discrepancy is not clear. As in the case of silver, the rate of solution of gold is controlled by the diffusion of one of the reactants ($CN^-$ or $O_2$) to the surface; the apparent activation energy is 3 to 5 kcal per mol.

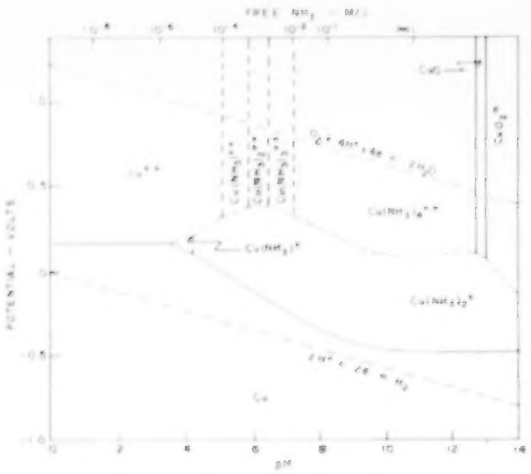

Fig. 2—Potential *pH* diagram for the Cu-NH$_3$-H$_2$O system. Total NH$_3$ equals 1 mol per liter.

The kinetics of dissolution of silver in acid solutions containing various oxidants have also been determined. When $Ce^{++++}$ was employed as oxidant, its diffusion to the silver surface was rate-limiting,[29] while with $Fe^{+++}$ the rate was chemically controlled.[30] This has led to the conclusion[29] that, in general, metal dissolution reactions with a large potential difference between the metal and the oxidant are diffusion-controlled, while those with a small potential difference are chemically (i.e., desorption) controlled. Because of the great stability of the silver-cyanide and gold-cyanide complexes, the potentials for the dissolution of gold and silver are so much lowered by the presence of cyanide that the cyanidation reactions apparently fall into the former category.

**Leaching of Copper**—The thermodynamics of this system have been discussed earlier and are summarized in the form of potential-*pH* diagrams in Figs. 1 and 2.

The reaction involving acid solutions in the presence of oxygen is

$$Cu + 1/2\,O_2 + 2\,H^+ \rightarrow Cu^{++} + H_2O \qquad [12]$$

The kinetics in sulfuric acid solution were found by Lu and Graydon[31] to conform to

$$\text{rate} = k\,[Cu^{++}]^{0.5}[O_2]^{1.0} \qquad [13]$$

the apparent activation energy being 14.1 kcal per mol.

The proposed mechanism involves the following sequence of steps

$$Cu^{++} + Cu \rightleftharpoons 2Cu^{+} \qquad [14a]$$

$$Cu^{+} + H^+ + O_2 \underset{\text{slow}}{\rightarrow} Cu^{++} + OH \qquad [14b]$$

$$Cu^{+} + OH \rightarrow Cu^{++} + OH^- \qquad [14c]$$

$$OH + H^+ \rightarrow H_2O \qquad [14d]$$

This mechanism is consistent with the observed kinetics if reaction 14b is assumed to be rate-determining.

The following kinetics were observed in HCl solutions[31]

$$\text{rate} = \frac{k_c k_a [H^+][O_2]}{1 + (k_c/k_a^-)[H^+] + K_a[O_2](K_b[H^+][Cl^-])} \qquad [15]$$

where the various equilibrium constants, $K$, and rate constants, $k$, refer to the following reaction sequence.

$$2Cu + O_2 \rightleftharpoons Cu_2{\cdot}O_2\,(\text{adsorbed}) \qquad [16a]$$

$$Cu_2{\cdot}O_2\,(\text{adsorbed}) + HCl\ (\text{or } H^+ + Cl^-) \rightleftharpoons Cu_2{\cdot}O_2{\cdot}HCl\,(\text{adsorbed}) \qquad [16b]$$

$$Cu_2{\cdot}O_2\,(\text{adsorbed}) + H^+ \underset{\text{slow}}{\rightarrow} 2Cu^{+} + HO_2^- \qquad [16c]$$

$$2Cu^{+} + HO_2^- + 3H^+ \rightarrow 2Cu^{++} + 2H_2O \qquad [16d]$$

Displaying an unusual effect, $Cl^-$ inhibits the dissolution of copper by competing for surface sites in reaction 16b.

Dissolution of copper is generally much more rapid in the presence of $NH_3$ and $NH_4^+$, where stable complex ions of $Cu^{++}$ are formed according to the reactions

$$Cu + 1/2\,O_2 + 4NH_3 + H_2O \rightarrow Cu(NH_3)_4^{++} + 2OH^- \qquad [17]$$

or

$$Cu + 1/2\,O_2 + 2NH_3 + 2NH_4^+ \rightarrow Cu(NH_3)_4^{++} + H_2O \qquad [18]$$

Several kinetic investigations have been reported[32,33,34] on this system. At low values of the ratio $[O_2] : [Cu(NH_3)_4^{++}]$ the reaction is autocatalytic,[32] corresponding to the following kinetics

$$\text{rate} = k\,[Cu(NH_3)_4^{++}]^{1.0}V^{0.5} \qquad [19]$$

where $V$ is the stirring velocity. The apparent activation energy is 2.9 kcal per mol. The proposed mechanism[32] assumes that the equilibrium

$$Cu(NH_3)_4^{++} + Cu \rightleftharpoons 2\,Cu(NH_3)_2^{+} \qquad [20]$$

is rapidly established and that the rate-determining process is the removal (by diffusion) of $Cu(NH_3)_2^{+}$ from the surface to the bulk of the solution, where it is presumably oxidized rapidly to $Cu(NH_3)_4^{++}$.

At high values of $[O_2]$ and of the ratio $[O_2] : [Cu(NH_3)_4^{++}]$ (corresponding to region $B$ in Fig. 4), the kinetics are given by

$$\text{rate} = k_a[NH_3] + k_c[NH_4^+] \qquad [21]$$

where $k_c/k_a = 20$. Thus $NH_4^+$, which does not influence the thermodynamics of the leaching reaction, apparently exerts a powerful influence on the rate. The constants $k_a$ and $k_c$ are independent of $[O_2]$ and of $[Cu(NH_3)_4^{++}]$. The following mechanism has been proposed[33]

$$Cu + 1/2\,O_2 \rightarrow Cu_2{\cdot}O\,(\text{adsorbed}) \qquad [22a]$$

$$Cu_2{\cdot}O\,(\text{adsorbed}) + NH_3 + H_2O \underset{\text{slow}}{\rightarrow} Cu(NH_3)_2^{++} + 2\,OH^- \qquad [22b]$$

$$Cu_2{\cdot}O\,(\text{adsorbed}) + NH_4^+ \underset{\text{slow}}{\rightarrow} Cu(NH_3)_2^{++} + OH^- \qquad [22c]$$

$$Cu(NH_3)_2^{++} + 3\,NH_3 \rightarrow Cu(NH_3)_4^{++} \qquad [22d]$$

This mechanism assumes that the copper surface is completely covered by a film of adsorbed oxygen (or

by a thin layer of copper oxide) which is formed rapidly in reaction 22a. Attack on this film by $NH_4$ (reaction 22b), or $NH_3$ (reaction 22c), is the rate-determining step in the dissolution process. The higher rate constant for $NH_3$ probably reflects its ability to attack the copper and oxygen surface sites simultaneously

$$Cu\ldots O + NH_4^+ \rightarrow \left[\begin{matrix} Cu\ldots NH_3 \\ \vdots \quad\quad \vdots \\ O\ldots H \end{matrix}\right]^+ \rightarrow Cu(NH_3)^{++} + OH^- \qquad (23)$$

INTERMEDIATE

This represents a phenomenon which is fairly generally encountered, i.e., metal oxides are often more readily attacked by an undissociated acid molecule, $HX$ (particularly when $X^-$ is capable of forming a complex ion of the metal) than by either $H^+$ or $X^-$ separately.

Alloying copper with small amounts of gold (which does not dissolve in ammonia) lowers its rate of solution markedly.[48] This is due partly to the protective influence of the inert gold-rich phase which is left behind as the copper dissolves and partly to the fact that favorable conditions are created for the formation of passivating deposits of copper oxide.

**Leaching of Oxides**—An understanding of the factors which influence the solution of oxides is important not only because of the intrinsic interest of these systems but also because it is probable that oxides (or compounds resembling oxides) are often formed as intermediates during the leaching of metals and metallic sulfides. A number of oxide leaching reactions have been studied.

Pryor and Evans[49] investigated the decomposition of ferric oxide by aqueous solutions of various acids. Hydrated ferric oxide dissolves readily in most acids. The $\alpha$-ferric oxide, prepared by ignition above 300°C, is readily dissolved by a reductive mechanism, but direct dissolution by the reaction

$$Fe_2O_3 + 6\,H^+ \rightarrow 2\,Fe^{+++} + 3\,H_2O \qquad [24]$$

proceeds only slowly even under very favorable thermodynamic conditions. Acids whose anions form complexes with $Fe^{+++}$ decompose the oxide more rapidly than those of non-complexing anions. Thus the rate of solution in different acids decreases in the order $HF > HCl > H_2SO_4 > HClO_4$. The rate of solution of ferric oxide by HCl increases with the HCl concentration but is unaffected by addition of neutral $Cl^-$ salts. The mechanism of dissolution probably involves simultaneous attack of $H^+$ and $Cl^-$ (presumably at oxygen and iron sites respectively). The attack apparently occurs only in the vicinity of surface lattice defects which are formed during ignition of the oxide, rather than uniformly over the whole surface.

Wadsworth and Wadia[50] have examined the decomposition of cuprite by sulfuric acid solutions. The reaction, involving disproportionation of Cu(I), is represented by

$$Cu_2O + 2\,H^+ \rightarrow Cu^{++} + Cu^0 + H_2O \qquad [25]$$

The rate, which depends on the concentration of both $H^+$ and undissociated $H_2SO_4$, is given by

$$\text{rate} = \frac{K_a\,[H_2SO_4]\,(k_b + k_c\,[H^+])}{1 + K_a\,[H_2SO_4]}. \qquad [26]$$

The following mechanism has been proposed to explain the kinetics:

$$Cu_2O + H_2SO_4 \rightleftharpoons Cu_2O{\cdot}H_2SO_4\ \text{(adsorbed)} \qquad [27a]$$

$$Cu_2O{\cdot}H_2SO_4\ \text{(adsorbed)} \xrightarrow[\text{slow}]{} Cu^{++} + Cu^0 + H_2O + SO_4^= \qquad [27b]$$

$$Cu_2O{\cdot}H_2SO_4\ \text{(adsorbed)} + H^+ \xrightarrow[\text{slow}]{} Cu^{++} + Cu^0 + H_2O + HSO_4^- \qquad [27c]$$

The reaction may occur either through reactions 27b or 27c, the rates being additive.

The kinetics of the oxidative leaching of pitchblende by oxygen-containing carbonate-bicarbonate solutions have been investigated.[51] The reaction is

$$U_3O_8 + 1/2\,O_2 + 3\,CO_3^= + 6\,HCO_3^- \rightarrow 3\,UO_2(CO_3)_3^{----} + 3\,H_2O. \qquad [28]$$

The kinetics are given by

$$\text{rate} = k\,[O_2]^{1/2}. \qquad [29]$$

The $k$ is nearly independent of the concentrations of $CO_3^=$ and $HCO_3^-$, providing these concentrations exceed certain critical values. The apparent activation energy is about 12 kcal per mol. By application of the absolute reaction-rate theory, the kinetics were shown to be consistent with the following mechanism:

$$U_3O_8 + 1/2\,O_2 \xrightarrow[\text{slow}]{} 3\,UO_3 \qquad [30a]$$

$$UO_3 + CO_3^= + 2\,HCO_3^- \rightarrow UO_2(CO_3)_3^{----} + H_2O. \qquad [30b]$$

In this case, the initial oxidation reaction 30a appears to be rate-determining; dissolution of the oxidized surface product follows rapidly.

**Leaching of Sulfides**—The chemistry of the leaching of metal sulfides is usually more complicated than that of metals or oxides and a greater variety of products may be encountered. Hence progress toward the understanding of such systems has been more limited.

One of the first reactions to be investigated kinetically was the leaching of galena by oxygen-containing sodium hydroxide solutions.[52]

$$PbS + 2\,O_2 + 3\,OH^- \rightarrow HPbO_2^- + SO_4^= + H_2O. \qquad [31]$$

This reaction proceeds with moderate rates at temperatures of 100° to 180°C, $O_2$ partial pressures of 2 to 10 atm, and NaOH concentrations of 0.5 to 6 mol per liter. Its study is simplified by the fact that all the products are soluble and hence the galena

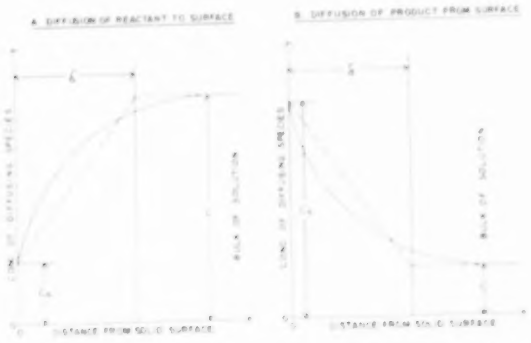


Fig. 3—Solution concentration of a diffusing species in the vicinity of a reacting solid surface.

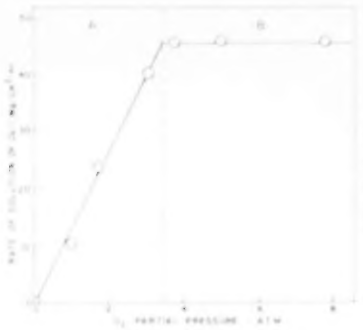

Fig. 4—Dependence of the rate of solution of copper in 0.74 mol $NH_3$ on oxygen concentration at 26°C.

surface does not change chemically during leaching. The rate appears to be chemically determined and has an apparent activation energy of 6.3 kcal per mol. The kinetics were analyzed in terms of the absolute-rate theory and the following mechanism was originally proposed:[33]

$$PbS + 1/2\,O_2 \rightleftharpoons PbS \ldots O\ (adsorbed) \quad [32a]$$

$$PbS \ldots O(adsorbed) + H_2O \xrightarrow{slow} \bullet$$

$$PbS{\cdot}(OH)_2(adsorbed) \quad [32b]$$

$$PbS{\cdot}(OH)_2(adsorbed) + 1\,1/2\,O_2 + 3\,OH^- \rightarrow HPbO_2^- + SO_4^{=} + H_2O \quad [32c]$$

This mechanism was intended to account for the insensitiveness of the rate to the $OH^-$ concentration and its apparent 0.5 order dependence on the $O_2$ concentration. However, it has been pointed out[34] that the results are equally well fitted by a model involving adsorption of molecular $O_2$ (i.e., non-dissociative adsorption) if a greater degree of surface coverage is assumed. In view of the large valence change involved, the mechanism by which the sulfur is oxidized from the sulfide to sulfate form is probably fairly complex.

Under comparable conditions, lead can also be leached from galena by neutral ammonium acetate solutions[35] according to the reaction

$$PbS + 1/2\,O_2 + 2\,NH_4Ac \rightarrow PbAc_2 + S^{\circ} + 2\,NH_4 + H_2O \quad [33]$$

The liberated sulfur appears in elemental form, as a film which covers the galena surface and isolates it from the solution. The parabolic kinetics[35] suggest that diffusion through this film is rate-limiting, although the nature of the diffusing species has not been established.

A kinetic study of the leaching of molybdenite by alkaline solutions

$$MoS_2 + 1/2\,O_2 + 6\,OH^- \rightarrow MoO_4^{=} + 2\,SO_4^{=} + 3\,H_2O \quad [34]$$

has recently been described.[36] This reaction is superficially similar to the leaching of galena, Eq. 31, and proceeds under comparable conditions, i.e., at temperatures of 100° to 175°C. However, the kinetics are appreciably different, particularly in respect to the dependence of the rate on the concentration of $OH^-$. In this case a sulfur compound of intermediate valence, $S_2O_3^{=}$, could be identified in the solution. To explain the kinetic results, a mechanism was proposed[36] in which oxygen adsorption, followed by slow attack of $OH^-$, is rate-determining.

A kinetic study has been made[37] of the leaching of pyrite by alkaline solutions under oxygen pressure. The chemistry of the system is very complex. The final products comprise a soluble sulfate salt and a mixture of iron oxides, notably $Fe_3O_4$ and $Fe_2O_3$. The iron oxides form a stable layer around the sulfide particles. The results are qualitatively consistent with a model in which the diffusion of sulfur and oxygen in opposite directions through the oxide layer determines the rate.

It is probable that similar considerations apply to the ammonia leaching of pentlandite under oxygen pressure, a process now being used commercially to extract nickel, along with copper and cobalt, from sulfide concentrates.[38] It has been suggested[39] that the principal reaction is

$$NiS{\cdot}FeS + 3\,FeS + 7\,O_2 + 10\,NH_4 + 4\,H_2O \rightarrow Ni(NH_3)_4SO_4 + 2\,Fe_2O_3{\cdot}H_2O + 2\,(NH_4)_2S_2O_3 \quad [35]$$

followed by oxidation of the $(NH_4)_2S_2O_3$ in solution. Diffusion of soluble reactants and products through the insoluble layers of iron oxide which coat the sulfide particles appears to be among the most important rate-determining factors. Adsorption of $Ni^{++}$ and $Co^{++}$ by the iron oxide may be important in limiting the ultimate recovery of these ions. The system is one of great complexity and many features of its chemistry remain to be elucidated.

The oxidation of iron sulfides has also been studied in neutral and acid solutions.[40] At temperatures of 100° to 125°C, and moderately high oxygen pressures, pyrrhotite reacts to form elemental sulfur in good yield

$$2\,FeS + 3\,O_2 \rightarrow 2\,Fe_2O_3 + 2\,S^{\circ} \quad [36]$$

While the kinetics of the reaction have not been investigated in detail, the following mechanism has tentatively been proposed to explain the results

$$FeS + 2\,O_2 \rightarrow FeSO_4 \quad [37a]$$

$$6\,FeSO_4 + 1\,1/2\,O_2 \rightarrow 2\,Fe_2(SO_4)_3 + Fe_2O_3 \quad [37b]$$

$$Fe_2(SO_4)_3 + 3\,H_2O \rightarrow Fe_2O_3 + 3\,H_2SO_4 \quad [37c]$$

$$FeS + H_2SO_4 \rightarrow FeSO_4 + H_2S \quad [37d]$$

$$2\,H_2S + O_2 \rightarrow 2\,H_2O + 2\,S^{\circ} \quad [37e]$$

$$H_2S + Fe_2(SO_4)_3 \rightarrow 2\,FeSO_4 + H_2SO_4 + S^{\circ} \quad [37f]$$

This mechanism is intended to account for the fact that no elemental sulfur is observed to form unless a solution *pH* of below about 1.5 is established. At temperatures below 130°C only about 10 to 15 pct of the sulfur is oxidized to sulfate form.

The oxidation of pyrite under the same conditions yields hardly any elemental sulfur, but results almost exclusively in the formation of sulfate. It has been suggested that the following are among the principal contributing reactions:

$$2\,FeS_2 + 7\,O_2 + 2\,H_2O \rightarrow 2\,FeSO_4 + 2\,H_2SO_4 \quad [38a]$$

$$2\,FeSO_4 + H_2SO_4 + 1/2\,O_2 \rightarrow Fe_2(SO_4)_3 + H_2O \quad [38b]$$

$$Fe_2(SO_4)_3 + (3 + x)H_2O \rightleftharpoons Fe_2O_3{\cdot}x\,H_2O + 3\,H_2SO_4 \quad [38c]$$

$$Fe_2(SO_4)_3 + (2 + 2x)H_2O \rightleftharpoons 2\,Fe(OH)SO_4{\cdot}x\,H_2O + H_2SO_4 \quad [38d]$$

The kinetics and mechanisms of these reactions remain to be elucidated. Also, it is not clear why elemental sulfur fails to form in this system.

Of interest is a recent kinetic study of the oxidation of sulfide minerals by aqueous chlorine solutions.[41] The reactions are faster than the correspond-

ing reactions involving oxygen and may be studied at much lower temperatures and pressures. However, the results parallel those obtained with oxygen in that pyrite, which is oxidized very rapidly by a diffusion-controlled reaction, yields only sulfate, while pyrrhotite and galena generally react to yield elemental sulfur. An explanation for this has been suggested which attributes the formation of elemental sulfur to the hydrolysis of a sulfur chloride intermediate.

Certain metal ions, notably $Cu^{++}$, are known to exhibit catalytic activity in a variety of oxidation-reduction reactions, including the reactions of molecular $O_2$ with compounds such as $Fe^{++}$[46] and $U^{++++}$.[47] There is good reason to believe that such ions, which are often present in leach solutions, may exert an important catalytic influence on the various homogeneous and heterogeneous reactions which occur during leaching, particularly of sulfides, thus affecting not only the leaching rates but also the nature of the final products. The investigation of this phenomenon by kinetic methods would appear to offer promising possibilities, but so far little work has been done in this direction.

## Precipitation Reactions

**Chemical Precipitation**—Chemical precipitation, involving the formation of insoluble hydroxides (by neutralization), sulfides, or other insoluble salts, is generally fast, since usually only simple ionic reactions are involved. The limitations in such processes are thus frequently of a thermodynamic nature. There are two important classes of exceptions.

1) When the substance being precipitated is present in low concentration, even though its normal solubility is exceeded, precipitation may be slow because of the tendency for a supersaturated solution to persist. In such cases, seeding the solution with precipitates is helpful.

2) Substitution reactions involving complex ions (including hydrolytic complexes), particularly of metals of high valence such as V(V) or U(VI) are often slow. Important examples are the precipitation of vanadic acid by neutralization of a vanadate solution[48]

$$5\ VO_3^- + 11\ H^+ \rightarrow H_4V_5O_{15} + 4\ OH^- \qquad [39]$$

and the precipitation of sodium uranate from a carbonate solution by addition of caustic soda[6]

$$UO_2(CO_3)_3^{----} + 2\ Na^+ + 4\ OH^- \rightarrow Na_2UO_4 + 3\ CO_3^{--} + 2\ H_2O \qquad [40]$$

Reactions such as these may take many hours to go to completion. Similarly, while most ammine complexes are readily decomposed by addition of acid, some like $Co(NH_3)_6^{+++}$ dissociate only very slowly. In many cases, the slow rates reflect the strong covalent bonds between the metal ion and the complexing ligand. For a comprehensive discussion of the rates and mechanisms of the substitution reactions of metal complex ions, the reader is referred to Taube's excellent review.[49]

**Precipitation of Gold from Cyanide Solutions by Zinc**—The physical-chemical factors influencing the precipitation of gold from cyanide solutions by zinc have recently been investigated by Hancock and Thomas.[50] They showed that the overall reaction

$$2\ Au(CN)_2^- + Zn \rightarrow 2\ Au + Zn(CN)_4^{--} \qquad [41]$$

may be considered as an electrochemical process involving separate anodic and cathodic steps as follows

$$\text{Anodic process:} \quad Zn + 4\ CN^- \rightarrow Zn(CN)_4^{--} + 2e \qquad [42]$$

$$\text{Cathodic process:} \quad Au(CN)_2^- + e \rightarrow Au + 2\ CN^- \qquad [43]$$

The efficiency of gold precipitation is regulated by a competing cathodic reaction involving liberation of $H_2$

$$2\ H_2O + 2e \rightarrow H_2 + 2\ OH^- \qquad [44]$$

The potential of this reaction is similar to that for the reduction of gold. Therefore, to minimize its effect, it is important that the hydrogen overpotential be kept as high as possible.

The presence of ions such as $Ni^{++}$ is often particularly detrimental to gold precipitation, since metallic nickel, which may be precipitated on the zinc surface, has a very low hydrogen overpotential. Conversely, salts of metals with high hydrogen overpotentials, such as lead, increase the efficiency of gold precipitation, as do addition agents such as gelatin. The detrimental effect of lime on gold precipitation also finds explanation in terms of this model.

**Reduction by Hydrogen**—The ability of gaseous hydrogen to reduce many dissolved metal salts provides a powerful method of recovering these metals from solution, either in metallic form or as insoluble compounds of lower valence. A number of processes of commercial interest have been developed using this procedure.

One of the applications first reported relates to the precipitation of metallic nickel from ammoniacal nickel sulfate solutions.[51-53] The reaction proceeds most conveniently in a solution in which the ratio of concentrations of $NH_3$ to $Ni^{++}$ is close to two, most of the nickel being present as the diammine complex, $Ni(NH_3)_2^{++}$. Under these conditions the overall reaction is

$$Ni(NH_3)_2^{++} + H_2 \rightarrow Ni^0 + 2\ NH_4^+ \qquad [45]$$

If a higher starting ratio of $NH_3$ to $Ni^{++}$ is employed, higher nickel ammine complexes are formed (particularly as the reaction proceeds and excess $NH_3$ is liberated) whose reduction by hydrogen is less favorable both from a thermodynamic and a kinetic standpoint than that of the diammine. A lower starting ratio results in free $H^+$ being generated by the reaction with adverse effects on the thermodynamics.

The reaction does not appear to proceed homogeneously in solution under the conditions usually employed (about 150° to 200°C and up to 400 psi $H_2$), but only on the surface of the metallic nickel which is already present, usually in the form of a suspended powder. This reflects the well known ability of metallic nickel to activate molecular hydrogen heterogeneously, a property responsible for its catalytic activity in many hydrogenation reactions. Nickel is thus an ideal metal to reduce with hydrogen, since the reaction is autocatalytic. To initiate the reaction, it is necessary either to add some metallic nickel powder, or to add a suitable catalyst such as a ferrous salt, which induces nucleation, i.e., the formation of very finely divided nickel particles. Once such particles are present, they serve as the seed for the subsequent heterogeneous growth reaction. The growth pattern of these particles has been investigated[53] and it has been established that beyond the first stages of growth the number of particles remains substantially constant

and that the rate of reaction at any subsequent time is directly proportional to the total surface area of the nickel particles present (as determined by an adsorption method). The effects of temperature and hydrogen pressure on the kinetics have been investigated[44] but more work remains to be done on these and other chemical variables before the detailed mechanisms of either the nucleation or growth reactions can be established.

The properties of cobalt are very similar to those of nickel and it is probable that considerations similar to those discussed above govern the precipitation of metallic cobalt from ammoniacal cobalt sulfate solutions in the absence of added catalysts. By suitable adjustment of conditions, to take advantage of a number of thermodynamic and kinetic factors, it is possible to achieve a high degree of separation of nickel and cobalt, starting with a solution containing both metals, by selectively precipitating them with hydrogen in separate stages.[45]

While metallic copper can also be precipitated by reducing cupric salt solutions with hydrogen, it is distinguished from nickel and cobalt by two important considerations.

1) Thermodynamically, cupric salts are much more readily reducible than either nickel or cobalt salts and hence metallic copper can be precipitated with hydrogen from either ammoniacal or acid solution (see Figs. 1 and 2).

2) Unlike nickel and cobalt salts, cupric salts can react homogeneously with hydrogen in aqueous solution under certain conditions and hence no catalyst is required to initiate the reaction.

The kinetics of the initial homogeneous reaction between $Cu^{++}$ and $H_2$ have been investigated in some detail[46-48] and are represented at moderate acidities by

$$-d[H_2]/dt = k[Cu^{++}][H_2] \quad [46]$$

where

$$k = 1.0 \times 10^{11} \exp[-26{,}600/RT] \text{ liter mol}^{-1} \text{ sec}^{-1}. \quad [47]$$

It is probable that the formation of metallic copper proceeds through the following sequence of steps:

$$Cu^{++} + H_2 \xrightarrow[\text{slow}]{k} CuH^{++} + H^+ \quad [48a]$$

$$CuH^{++} + Cu^{++} \rightarrow 2\,Cu^{+} + H^+ \quad [48b]$$

$$2\,Cu^{+} \rightarrow Cu^{++} + Cu^{0}. \quad [48c]$$

At high *pH*, such as in acetate buffered solutions, the disproportionation reaction 48c is replaced by another reaction[47,48] which leads to the formation of cuprous oxide instead of metallic copper, especially at low temperatures:

$$2\,Cu^{+} + 2\,OH^- \rightarrow Cu_2O + H_2O. \quad [49]$$

Complexes of $Cu^{++}$ with certain anions such as $SO_4^{--}$, $Cl^-$, and $Ac^-$ react more readily with $H_2$ than the uncomplexed $Cu^{++}$ ion, while ammine type complexes[49] probably react more slowly. Because of their ability to activate hydrogen, cupric salts can also function as homogeneous catalysts for the hydrogenation of other compounds, such as $Cr_2O_7^{--}$ in aqueous solution.[46,50,51]

Among the few other metal ions which share with $Cu^{++}$ the ability to react homogeneously with $H_2$ in aqueous solution under moderate conditions are $Ag^+$,[46] $Hg^{++}$,[52,53] $Hg_2^{++}$,[52] and $MnO_4^-$.[54]

The use of nonmetallic catalysts in hydrogenation reactions involving the reduction of dissolved metallic salts offers a particularly interesting and promising approach. Thus Kaneko and Wadsworth[55] have shown that colloidally dispersed graphite, or certain dissolved organic compounds such as hydroquinone, catalyze the reduction of ammoniacal cobalt sulfate in aqueous solution to metallic cobalt. The kinetics suggest that the function of the catalyst is to activate the hydrogen molecule and enable it to form an active hydrogen-carrying intermediate, $Co(II) \cdot 2H$, which decomposes rapidly to form metallic cobalt. The analogy between this mechanism and that proposed earlier for the reaction between $Cu^{++}$ and $H_2$, Eq. 48, is readily apparent.

A number of systems are known in which hydrogenation of a dissolved metal salt results in the precipitation of an insoluble oxide of lower valence.

1) Precipitation of cuprous oxide from a solution of cupric acetate[56]

$$2\,CuAc_2 + H_2 + H_2O \rightarrow Cu_2O + 4\,HAc. \quad [50]$$

2) Precipitation of uranium oxide from a uranyl carbonate solution[57]

$$UO_2(CO_3)_3^{----} + H_2 \rightarrow UO_2 + CO_2 + 2HCO_3^-. \quad [51]$$

3) Precipitation of vanadium oxide from a vanadate solution[58]

$$2\,VO_3^- + 2\,H_2 \rightarrow V_2O_3 + 2\,OH^- + H_2O. \quad [52]$$

All these reactions have been studied kinetically. The first occurs homogeneously and does not require a catalyst.[56] The last two reactions require the presence of a catalyst such as finely divided metallic nickel.

## Conclusions

In this paper an attempt has been made to discuss some of the thermodynamic and kinetic factors which influence hydrometallurgical processes and whose understanding is important in the long run to the efficient operation and control of such processes. A number of specific reactions have been discussed in some detail. The list of these was intended to be selective rather than comprehensive, and the task of selection was greatly facilitated by the fact that of the great number of known reactions of hydrometallurgical interest only a few have been subjected to sufficiently systematic investigation to permit elucidation of their physical chemistry. Preference was given to the review of recent work and to the consideration of subject matter and ideas which seem to the author to have an important place in the future development of the subject as a whole.

In general, our knowledge about the thermodynamics of hydrometallurgical reactions is in a fairly advanced state. The principles involved are well understood and a sufficient body of thermodynamic data is readily available in tabulated form to permit thermodynamic calculations to be made for most systems to a satisfactory degree of approximation. Unfortunately, thermodynamic data for aqueous solutions are usually available only for room temperature and there is a need for more reliable data at higher temperatures, particularly above 100°C. More data on the activities of salts in concentrated solutions and on the thermodynamic properties of complex ions would also be helpful. However, there is an encouraging amount of interest and activity in this field and it is reasonable to expect that such

gaps as now exist in our knowledge will continue to be filled at a satisfactory rate.

Our understanding of the kinetics and mechanisms of most hydrometallurgical reactions is in a much less satisfactory state, an unfortunate situation in view of the fact that the application and efficiency of many hydrometallurgical processes appear to be limited by kinetic considerations. There is a great need for more careful and systematic work on the kinetics of many reactions, and the importance of careful design of experiments and control of variables if an investigation is to yield useful information can hardly be emphasized sufficiently. Often the conditions under which a reaction must be studied in order to derive the greatest amount of information (including practical information) are far removed from those under which it is conducted commercially, and failure to appreciate this has resulted in the expenditure of much wasted effort in the past. For example, in studying the leaching of a solid, it is usually preferable, where possible, to use large specimens whose surface area can be accurately determined and held constant during an experiment, rather than a pulp of finely divided material whose surface area and variation as leaching proceeds are usually very uncertain. It is also very important, in studying heterogeneous reactions, for the effect of agitation to be carefully examined in each case and, if possible, for the reaction to be studied under conditions where the rate is free from transport control.

Where suitably designed kinetic experiments have been performed and the necessary kinetic data obtained, application of the absolute reaction rate theory[66] has permitted the elucidation of the reaction mechanism, sometimes even in fairly complex systems. Most of the work which has been done along these lines is reviewed in this paper. One of the striking impressions which emerges is that each of the reactions which has been studied is highly individual in character. Even in a group of superficially related reactions such as the oxidative leaching of metals or of metallic sulfides, the differences in kinetics and mechanism between individual reactions are often more significant than the similarities. This suggests that many more processes will have to be studied in detail and their mechanisms elucidated before it is possible to advance any generalizations or to make confident predictions about new systems.

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Technical Note

# Information from Normal Grain Growth

by C. G. Dunn and P. K. Koh

IN an investigation of the rolling and recrystallizing textures obtained from single crystals of Si-Fe, Koh and Dunn included specimens in the (111) [110] orientation.[1,2] This note reports some additional results obtained on samples of this orientation set aside for further study. After primary recrystallization longer annealing treatments at 980°C produced only normal grain growth; after 16 hr at 980°C the largest grains were approximately 0.010 to 0.020 in. in diam.

The orientations of 13 of the larger grains were found by the transmission Laue method. The unidentified {110} poles of these grains have been added to the {110} pole figure obtained after primary recrystallization[1] to give the plot shown in Fig. 1. When grain identifications are also included, such a plot reveals the following. Two orientations agree almost exactly with the peak pole density positions of component *B* of the primary recrystallization texture, one with component *A*, and one with component *C*. All the remaining orientations except one have {110} poles near weaker and unidentified pole density peaks. In fact, the correlation is good enough for an identification of eight components. Previously only components *A*, *B*, and *C* were certain.

The results are perhaps even more informative when combined with the {110} pole figure of the cold-rolled crystal to give the plot shown in Fig. 2. Each of the 13 orientations is identified and uniquely determined by selecting two {110} poles per crystal, which are 90° apart. The plot shows that every grain of the 13, except grain 6, has a {110} pole in common with a {110} pole concentration of the cold-rolled crystal. The orientation relationship with the cold-rolled orientation may be expressed as approximately 25° rotations about <110> axes, which is in agreement with the results found for coarse-grained primary recrystallization.[3] If primary recrystallization had occurred according to the <110> rule without preference, there would have been 12 components. Actually our limited data reveals nine of the possible 12. The integrated pole figure also shows preferences among them, since three are considerably stronger than the rest.

In addition to the above direct information, the following conclusions may be drawn. Primary recrystallization of the (111) [110] cold-rolled crystal produces a limited number of weak and strong components with the larger than average primary grains from these components related to the cold-rolled crystal by 25° <110> relationships. In arriving at this conclusion of limited orientations the (111) [110] orientation has to be ruled out as a source of large primaries for two reasons. First, growth in this orientation was not observed, despite an expected high grain boundary mobility (due to its 25° <110> relationship with every component of the texture). Secondly, primary recrystallization usually produces grains in new orientations, particularly in a single orientation matrix; but (111) [$\bar{1}$10] is not new. Other orientations besides those of the <110> type may be considered unlikely, of course, because the <110> relationship holds quite well for primary recrystallization[3] and should produce the large primaries. There are exceptions to this rule, but probably not enough to provide large

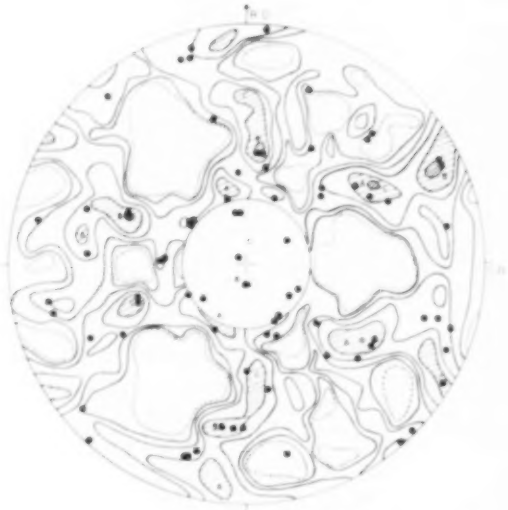

Fig. 1—{110} poles (closed circles) of 13 grains superimposed on the {110} pole figure obtained for primary recrystallization.

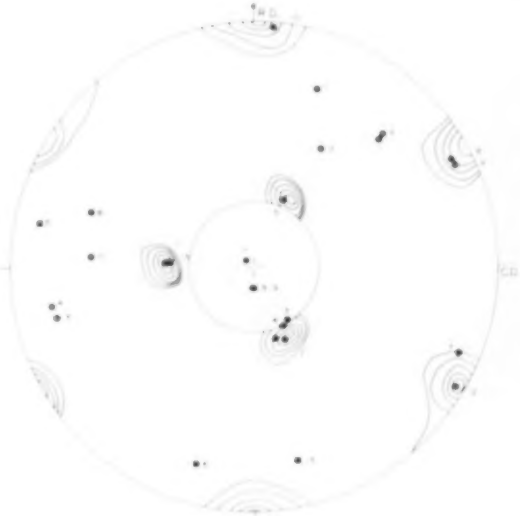

Fig. 2—{110} pole stereogram limited to two {110} poles per grain (identified by number) superimposed on the {110} pole figure of the cold-rolled crystal.

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primary grains in all orientations. If large primary grains are not present in all orientations, oriented growth theory[6] cannot be applied to explain the present results.

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Technical Note

# Preferred Orientation in Extruded Uranium Rod


by L. K. Jetter and C. J. McHargue


DEVELOPMENT of preferred orientation in uranium is of interest because of the strongly anisotropic properties of the orthorhombic crystal structure. Harris[1] reported inverse pole figures for rolled uranium rods which showed <010> and <410>* to be parallel to the rolling direction after hot-rolling. The relative strengths of the two components varied with the amount of reduction—the <010> component was stronger for a rod given a light reduction and the <410> component was stronger for a rod given a heavier reduction. Cold-rolling with a light reduction produced a strong concentration of <010> parallel to the rolling axis and a weaker concentration of <320>. Foote,[2] in a review paper, stated that early investigators reported textures having <010> and <410> parallel to the axis in rods produced by rolling at 300° and 600°C. The <010> component seemed to predominate at the lower temperature and <410> at the higher temperature. Mueller, Knott, and Beck at Argonne National Laboratory determined inverse pole figures by a modified Harris method which showed the maximum axis density to be near <031> for a reduction of 70 pct by rolling at 300°C. Rods rolled at 600°C after having been $\beta$-treated developed a strong <410> component with considerable spread toward the <010> and some spread toward <100>.[3]

Early investigators reported that the textures of swaged or rolled rods did not change after recrystallization;[4] however, Chernock and Beck[5] found definite textural changes occurring upon recrystallization. After an anneal at 575°C, the <410> component of a 300°C rolled rod was found to be replaced by a near <110> or <321> component and the retained <010> component was much decreased in intensity.

This note presents the results of a study on the textures of extruded rod which was carried out a few years ago.

Billets of uranium were extruded at 500°C to give rods of 1.5-in. and 0.9-in. diam (reductions in area of 77 pct and 91.7 pct, respectively). A portion of the 0.9-in. rod was annealed 1 hr at 550°C.

* Previous investigators have reported the fiber textures of uranium in terms of poles of planes parallel to the rod axis. In this paper these have been translated into indexes of direction.


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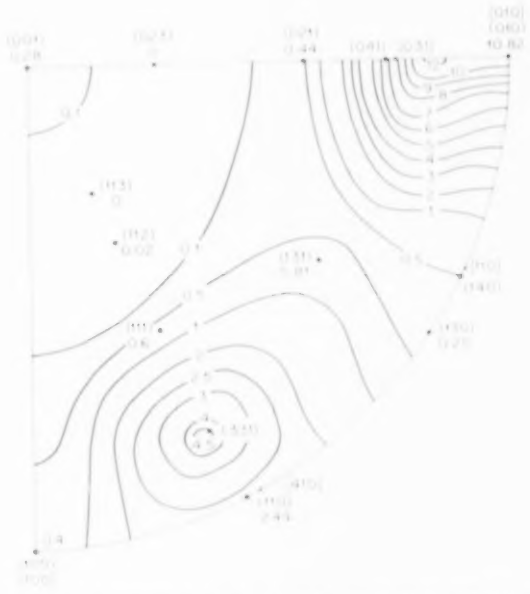

Fig. 1—Fiber axis distribution chart for uranium rod extruded at 500°C (reduction in area, 77 pct).

On 0.19-in. spherical X-ray diffraction specimens taken from the center of the middle section of each rod, pole distribution data were obtained by the spectrometric method described previously.[7] The specimen was rotated rapidly about the axis coinciding with the rod axis in order that a large number of grains could be scanned and was rotated slowly about an axis perpendicular to the rod axis. The variation in the diffracted X-ray intensity was recorded as a function of the latter motion (angle $\phi$).

Fiber-axis distribution charts (inverse pole figures) were prepared by the method described by Jetter, McHargue, and Williams.[8] After having been corrected for background radiation, the pole distribution data ($I$ vs $\phi$) were normalized. A normalizing factor for each plane has been shown to be $I_{\text{normal}} = \int_0^{\pi/2} I \sin\phi \, d\phi$ and was obtained by graphical integration from the data for that plane plotted as $I \sin\phi$ vs $\phi$. The axis-distribution charts deduced from the normalized pole densities are shown in Figs. 1 to 3. On these charts, the contours indicate angular positions of equal normalized axis density,

Fig. 2—Fiber axis distribution chart for uranium rod extruded at 500°C (reduction in area, 91.7 pct)

Fig. 3—Fiber axis distribution chart for uranium rod extruded at 500°C (reduction in area, 91.7 pct), and annealed 1 hr at 550°C.

and a value of unity corresponds to a random distribution.

The axis distribution chart for the rod given a reduction in area of 77 pct by extruding at 500°C shows a duplex texture (Fig. 1). Forty-six pct of the material is within 20° of the maximum which lies between <031> and <010> and 52 pct of the material has orientations within 20° of a peak which is 10° from <410> and 27° from <100>. The positions of peak intensity are approximately 10° from <010> and <410>. The microstructure of this specimen showed no evidence of recrystallization.

The axis distribution chart for the rod given a reduction in area of 91.7 pct is shown in Fig. 2. The maximum density is still at a position between <031> and <010>, but is now shifted toward <010>. Approximately 30 pct of the volume is associated with this component. The second peak, which is shifted toward <100> and <410>, is 7° from <410> and 21° from <100>. Approximately 70 pct of the volume is associated with this component. The microstructure of this specimen exhibited a number of small recrystallized grains.

Fig. 3 shows the axis distribution chart for the rod reduced 91.7 pct at 500°C and annealed 1 hr at 550°C. Quite obviously, recrystallization produced changes in the texture. The texture is duplex, having one component near <431> and a second component which is approximately 13° from <100> and 17° from <410>. Approximately 65 pct of the volume is associated with the former component and 35 pct with the latter.

None of the axis distribution charts was found to have the peak intensity at directions of low indexes. The previously reported components of <010> and <410> were found to lie 10° to 15° from those reported here. It is not unlikely that a quantitative treatment of the earlier data would show the components to be similar to those reported here. This is also indicated by the work of Mueller, Knott, and Beck reported by Foote,[2] who found the position of peak intensity in a rod rolled 70 pct at 300°C to be very near that reported for the as-extruded rods in this paper. The <110> or <321> annealing component reported by Chernock and Beck[3] may actually be similar to the recrystallized component which lies between these directions, namely <431>.

The present study indicates that the component near <031> decreases with increasing reductions with a corresponding increase in the near <410> component. In this case, increasing the reduction in area from 77 to 91.7 pct resulted in a decrease in the volume of material associated with the <031> component from 46 to 30 pct. These results are in somewhat qualitative agreement with the predictions by Calnan and Clews[6] that <010> → <320> directions should align with the rod axis for low amounts of deformation, and <410> for high amounts of deformation above 300°C.

## Summary

The deformation texture of uranium rod extruded at 500°C is duplex—the components being near <031> and 10° from <410>. Increasing amounts of deformation result in shifts from these positions toward <010> and <410>, respectively. The <031> component appears to be favored for low amounts of deformation and the near <410> component for high reduction.

The texture for uranium rod extruded at 500°C and annealed at 550°C is also duplex—one component near <431> and the other component being 13° from <100> and 17° from <410>.

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# AIME NEWS

## Technical Program Has Been Set for Regional Meeting in Detroit on Deep Drawing Steels

Donald Frey will coordinate activities as program chairman for the steel meeting.

A comprehensive program of 12 technical papers on the drawing quality of steels will be presented at a regional meeting in the Rackham Building, Detroit, March 18-19, sponsored by the Detroit Section and the AIME, NOHC.

On Monday morning, March 18, papers devoted to process metallurgy of deep drawing steels will include *Bottle Topped Steel*, by R. Hindson, Steel Co. of Canada; *Chemically Capped Steel* by J. S. McNairn, Ford Motor Co.; *Vanadium Killed Steels* by J. W. Frane, Bethlehem Steel Co.; and *Manufacture and Processing of Low-Carbon Rimming Steels for Deep-Drawing Applications* by E. M. Patterson, Inland Steel Co.

The afternoon session will include papers on *Effects of Hot Strip Mill Practices on the Quality of Deep Drawing Sheets* by D. Gottge and B. Southworth, U. S. Steel Corp.; and *Production Practices* by H. F. Lesso, Great Lakes Steel Corp.

The session on Tuesday morning, March 19, will be devoted to *Physical Metallurgy of Deep Drawing Steels* and includes papers on *Behavior of Interstitial Elements of Iron* by C. A. Wert, University of Illinois; *Boron Additions to Low-Carbon Steel* by E. R. Morgan, Jones & Laughlin Steel Co., and J. C. Shyne, Ford Motor Co.; and *Analytical Methods for Carbon Steels* by L. M. Melnick, U. S. Steel Corp.

That afternoon the theme, *Fabrication of Deep Drawing Steels*, will be carried out with papers on *Selection and Processing of Sheet Steel for Auto Body Panels* by H. Pratt, General Motors Corp.; *Application and Testing of Body Sheet* by F. M. Tousley, Chrysler Corp.; and *Some Factors in Physical Properties of Steel that Influence Deep Drawing Properties* by C. L. Altenburger, Great Lakes Steel Corp.

In addition to the technical program, a Social Hour and Banquet are planned for Monday evening.

### Welcoming Luncheon Speaker

Dr. J. C. Morris will be the featured speaker at the Welcoming Luncheon on Monday, Feb. 25, in the International Room, Hotel Roosevelt, during the Annual Meeting. His speech is provocatively titled *Around the World in 90 Minutes*.

A native of New Orleans, Dr. Morris received his B.S. and M.S. degrees from Tulane University, where he is head of the Dept. of Physics and has been vice president since 1947. He did graduate work at Princeton University, which awarded him the M.A. and Ph.D. degrees.

During World War II, he served as Director, Office of Scientific Personnel, National Research Council, and was also Assistant Director, anti-submarine laboratories, Office of Scientific Research and Development. In 1950 he was appointed by President Truman to the Board of the National Service Foundation for a four-year term; in 1954 he was reappointed for six years and is chairman of the Mathematical, Physical and Engineering Service Committee.

## To Address All-Institute Meeting in New Orleans

B. Brewster Jennings, Chairman of the Board, Socony Mobil Oil Co., N. Y., will be the featured speaker at the AIME All-Institute Technical Session Tuesday, Feb. 26, at 2:30 pm during the Annual Meeting in New Orleans. He will discuss one of the most pressing of current international problems—the Near East situation and how it affects world petroleum, in his talk, *Mankind's Stake in Middle East Oil*.

B. B. JENNINGS

A graduate of Yale, Mr. Jennings left school in 1917 to join the U. S. Navy and served overseas as an ensign on a submarine chaser. He returned to school in 1919, receiving his degree the following year.

Mr. Jennings has been associated with the company since 1920, beginning as a clerk in the marine department. Subsequently he became purchasing agent, and later manager of the real estate department. In 1935 he was appointed assistant to the president, and by 1939 was elected to the board of directors, in which capacity he supervised the company's transportation.

In 1942, Mr. Jennings left Socony to become Assistant Deputy Administrator for tanker operations of the

(Continued on page 361)

# Bylaws of the Metallurgical Society of AIME

Possible changes may be effected before final approval is given at the meeting of the AIME Board of Directors on Feb. 24, 1957.

## Article I

### Name and Object

**Section 1.** This unit of the American Institute of Mining, Metallurgical, and Petroleum Engineers Inc. shall be known as the Metallurgical Society of AIME.

**Section 2.** The object of the Society is to promote the advancement of metallurgical knowledge and the advancement of the metallurgical profession as a whole by:

**a.** Providing a medium for communication and cooperation among those interested in any phase of theoretical and applied metallurgy, including those advancing, practicing, learning, or otherwise primarily concerned with the science, engineering, economy, or technology of the metals industry (both producers and consumers). The major metallurgical fields encompass particularly the understanding and knowledge of metal behavior, the manufacturing practice for extracting, refining, and fabricating metals, and the development and application of metals and their alloys.

**b.** Providing an organization to represent the metallurgists and metallurgical engineers on matters pertaining to education, and to encourage and advance education in the broad field of metallurgical science and engineering.

**c.** Dissemination of metallurgical knowledge by the holding of professional meetings, and by the preparation, presentation, discussion, and publication of technical papers.

**d.** Promoting high standards in the profession of metallurgical engineering.

## Article II

### Members

**Section 1.** Any member of AIME in good standing may become a member of the Society by indicating his choice in writing to AIME headquarters. Members of the Society will receive the monthly journal(s) of the Society as part consideration for annual dues received. Only members in good standing in the AIME shall have privileges in the Society, and shall hold office in the Society and its Divisions.

## Article III

### Funds

**Section 1.** The expenditure of funds received by the Society shall be in conformity with rules established by the Executive Council of the Society.

**Section 2.** Dues or assessments in addition to the regular dues of AIME may be fixed by the Executive Council of the Society, subject to the approval of the Board of Directors of AIME.

**Section 3.** The Executive Council shall authorize the disbursement of funds for special purposes such as Awards, Scholarships, and Endowments.

**Section 4.** The officers of the Society shall submit an annual budget for approval of the Board of Directors of AIME, and shall operate the affairs of the Society in such manner as to assure a balanced budget as determined by AIME bookkeeping practices.

## Article IV

### Meetings

**Section 1.** The Society shall meet at the same time and place as the Annual Meeting of AIME and at such other times as may be determined by the Executive Council.

**Section 2.** The annual business meeting shall be held within a few days before or after the annual business meeting of the AIME.

**Section 3.** At a meeting of the Society, for which notice has been sent to the members of the Society through the regular mail or by publication in the Society's journal(s) at least one month in advance, a business meeting may be convened by order of the Executive Council, and any routine business may be transacted which is not inconsistent with these Bylaws or with the Constitution or Bylaws of the AIME.

**Section 4.** Twenty-five members of the Society shall constitute a quorum for the transaction of business at a regular constituted meeting.

## Article V

### Officers

**Section 1.** The officers of the Society shall consist of President, Vice President, Past-President, and Secretary-Treasurer.

**Section 2.** The Vice President shall be the President-Elect, and shall automatically succeed to the office of President at the end of his term as Vice President.

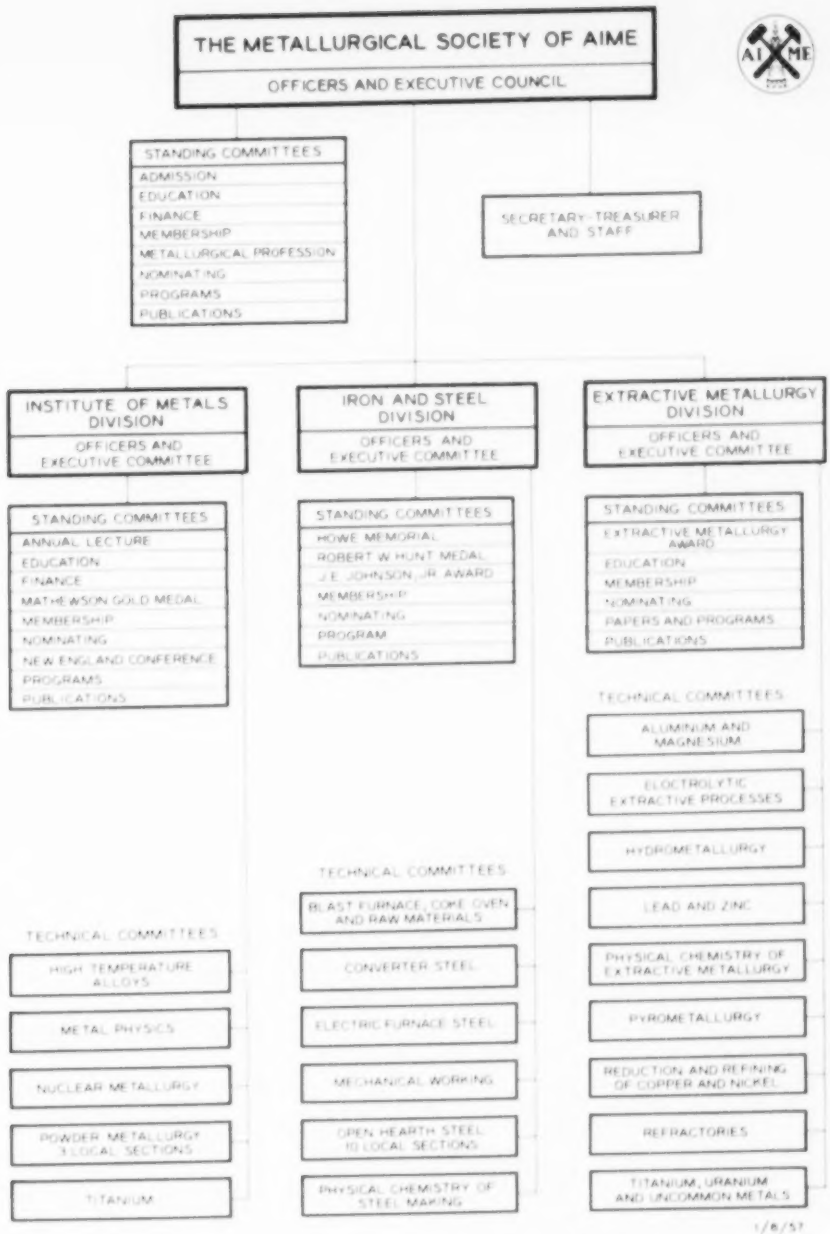
**Section 3.** The Executive Council shall consist of the following:

**a.** President
**b.** Vice President
**c.** Past-President
**d.** Secretary-Treasurer
**e.** Three representatives from each Division, one of whom shall be Division Chairman, provided that he not be serving as Vice President, President, or Past-President of the Society.

**Section 4.** The Secretary-Treasurer shall be appointed by the Executive Council for a term of one year at a salary consistent with his responsibilities, subject to the approval of the Board of Directors of AIME.

**Section 5.** The Society shall be represented on the Board of Directors of AIME by seven Directors as follows:

**a.** One member of the Society who will be currently serving as President-Elect, President, or Past-President of the Institute
**b.** Past-President of the Society




**c.** President of the Society
**d.** Vice President of the Society
**e.** Three Directors, elected for three-year terms
**f.** Should the Past-President, President, or Vice President of the Society be elected Past-President, President, or President-Elect of the Institute, the Society shall be represented by an additional Director to fill the unexpired term.

Each year, two of the Society's representatives on the AIME Board of Directors shall be designated as Vice Presidents of AIME, and shall serve for terms of one year each.

For an unexpired term, vacancies shall be filled by appointment by the President of the Society, subject to approval by the Society's Executive Council.

**Section 6.** The President-Elect and the representatives of this Society on the AIME Board of Directors shall be nominated and elected as provided hereafter in **Article VIII**, *Sections 1, 2, and 3.*

**Section 7.** The term of office for officers shall be one year; for other members on the Society's Executive Council three years; and for Society representatives on the AIME Board of Directors, three years. All terms of office shall start at the close of the annual business meeting of AIME.

## Article VI

### Divisions

**Section 1.** The Society shall consist of Divisions as established by the Executive Council, and initially comprise Extractive Metallurgy, Institute of Metals, and Iron and Steel. A member of the Metallurgical Society may belong to one or more Divisions of the Society by expressing his desire in writing to AIME Headquarters.

**Section 2.** Each Division shall be governed by its own officers and executive committee, and shall operate under its own bylaws. Bylaws of the Division Executive Committees shall be subject to approval by the Executive Council of the Society.

## Article VII

### Committees

**Section 1.** Standing Committees shall be appointed by the President, subject to confirmation by the Executive Council at each annual meeting of the Council. In general, appointments shall be made in each case for a specific term of years so that not all terms shall be completed in any one year.

**Section 2.** Special Committees may be established by the Executive Council as necessary or desirable. Members of Special Committees shall be appointed by the President, subject to confirmation by the Executive Council. Special Committees shall be held to a minimum in number, their duties shall be clearly defined, and they shall be dismissed as soon as their report is accepted.

**Section 3.** Standing Committees shall be:

- **a.** Finance Committee
- **b.** Programs Committee
- **c.** Publications Committee
- **d.** Membership Committee
- **e.** Education Committee
- **f.** Nominating Committee
- **g.** Committee on the Metallurgical Profession
- **h.** Admissions Committee

**Section 4.** The President shall appoint at the Annual Meeting of AIME a Finance Committee consisting of three of its members.

The Finance Committee shall prepare the annual budget, and examine, plan for, and submit recommendations concerning the financial condition and welfare of the Society for consideration of the Executive Council. The Secretary-Treasurer shall not be a member of the Finance Committee, but shall attend its meetings upon the Committee's request, and shall furnish such statements and information as the Committee may desire.

All bills, accounts, and claims against the Society within the approved budget of the year shall be examined and approved by the Secretary-Treasurer of the Society, or such officer or employee of the AIME as its Board of Directors shall from time to time designate.

**Section 5.** The Programs Committee shall consist of a Chairman appointed by the President and the chairmen of the Division Programs Committees. The Committee shall coordinate program plans and provide for such joint programs among the Divisions as are deemed desirable.

**Section 6.** The Publications Committee shall consist of at least six members of the Society, two from each Division, appointed by the President. The Committee shall determine publication policies of the Society, shall have authority to accept or reject papers to be published in the publication(s) of the Society, and shall have the duty of preparing and maintaining a publications budget.

**Section 7.** The Membership Committee shall consist of a Chairman appointed by the President and the Chairmen of the Division Membership Committees, and shall be responsible for the coordination of membership activities.

**Section 8.** The Education Committee shall be appointed by the President. This Committee shall devise a program to promote high standards in the education of metallurgists and metallurgical engineers, the execution of which is subject to the approval of the Executive Council.

**Section 9.** The Nominating Committee shall consist of three members from each Division, selected by each of the Division Nominating Committees, one of whom shall be selected as Chairman by the President.

**Section 10.** The Committee on the Metallurgical Profession shall be appointed by the President. This committee shall promote high standards in the profession of metallurgy and metallurgical engineering, and shall have the authority to cooperate with organizations having similar objectives.

**Section 11.** The Admissions Committee shall consist of a Chairman appointed by the President and two members from each of the Divisions of the Society. The Committee shall be responsible for reviewing the qualifications of candidates for membership and presenting the names of recommended candidates through the Secretary, AIME, to the Board of Directors of AIME for approval.

## Article VIII

### Nominations and Elections

**Section 1.** The Nominating Committee of the Society shall select:

- **a.** The nominee for Vice President (the President-Elect) of the Society, who automatically becomes a member of the AIME Board of Directors for a three-year term.
- **b.** Every third year, the nominee for President-Elect of AIME, who automatically serves a three-year term on the AIME Board of Directors successively as President-Elect, President, and Past-President.
- **c.** One nominee to serve as Director of AIME for a three-year term.
- **d.** Nominees for Directors of AIME to fill unexpired terms on the Board of Directors should a Director from the Metallurgical Society be selected as President-Elect of AIME or as Vice President (President-Elect) of the Society.

The Nominating Committee shall designate two Society members on the AIME Board of Directors or nominees for the Board of Directors to serve as Vice Presidents of the Institute for one-year terms.

If a previously elected Vice President of the Society should become unavailable for the Presidency of the Society, the Nominating Committee shall also designate a nominee for President.

**Section 2.** The names and biographies of nominees shall be published in the July issue of the publication(s) of the Society. Any 25 members of the Society may submit additional nominations to the Society's Executive Council for one or more of the positions specified in **Article VIII,** *Section 1.*

Such nominations shall be submitted not later than August 15 of each year, and notice of the additional nominees for the position designated shall then be published in the October issue of the publication(s) of the Society.

**Section 3.** If additional nominations are not received by August 15, the President of the Society shall declare the Nominating Committee's nominees to be elected. If additional nominations are received, a letter ballot showing all nominees for the positions designated shall be sent on or about October 1 to all members of the Society in the United States, Canada, and Mexico. The ballots shall be returned within 30 days after they are mailed. The incumbent President shall appoint a Tally Committee to count the ballots, and the nominees receiving the plurality of ballots for each position shall then be declared by the Executive Council to have been elected.

**Section 4.** Vacancies in any office of the Society occurring for any reason other than the expiration of term of election shall be filled by the President subject to approval by the Executive Council.

## Article IX

### Amendments

**Section 1.** Proposals to amend these Bylaws shall be made in writing to the Executive Council of the Society, and signed by at least ten members. These shall be considered by the Executive Council and announced to the members through the columns of the publication(s) of the Society, together with any comments or amendments made by the Executive Council thereon. Proposals to amend the Bylaws may also be made by the Executive Council. They shall be voted on at the Annual Meeting, or other meetings of the Council, or by letter ballot, as may be directed by the Executive Council. A majority of those voting is necessary to make the amendment. No proposal shall be submitted to members for voting that is inconsistent with the Constitution and Bylaws of AIME.

# AROUND THE SECTIONS

• For its first meeting of the 1956-1957 season, on Nov. 15, the **Cleveland Section** featured Harvey W. Maurice of Koppers Co. Inc., Pittsburgh, as guest speaker. After cocktails and dinner, Mr. Maurice addressed the group, assembled in the Hotel Manger, on *Some Recent Advances in Steelmaking*, emphasizing sintering, improvements in blast and open hearth furnaces, gas cleaning and direct casting. Mr. Maurice spent eight years with Union Carbide & Carbon Corp.'s National Carbon Div. where he was engaged in the application of graphite products to the steel industry. Since 1951 he has been associated with Koppers Co., correlating engineering, procurement and construction of sintering plants, blast furnaces and other steelmaking facilities.

• When the **El Paso** Metals Section met on Jan. 9 in the Mirror Room of Hotel Cortez, the guest speaker was Richard S. Smith, Phelps-Dodge Refining Co. Mr. Smith's talk was concerned with *Trends in Purchasing*.

• The **Chicago Section** met on Jan. 2, at the Chicago Bar Assn. at which time they heard Harold E. Rowen, general manager, Dwight-Lloyd Div., McDowell Co., Cleveland, who discussed *Some Developments in Pelletizing Jasper Concentrates*. Roy P. Wheatley, Blast Furnace Dept. Supt., Wisconsin Steel Works, presided. The Section's next meeting **Feb. 6**, featured F. M. Richmond, Research Head, Universal Cyclops Steel Corp., Bridgeville, Pa. Mr. Richmond's topic was *Properties of Vacuum Melted Materials*.

• The **Boston Section** held its Annual President's Night on Dec. 3, 1956. The guest of honor was AIME President C. E. Reistle, Jr. who was introduced to the members by L. W. Davis, Chairman of the Section's Executive Committee.

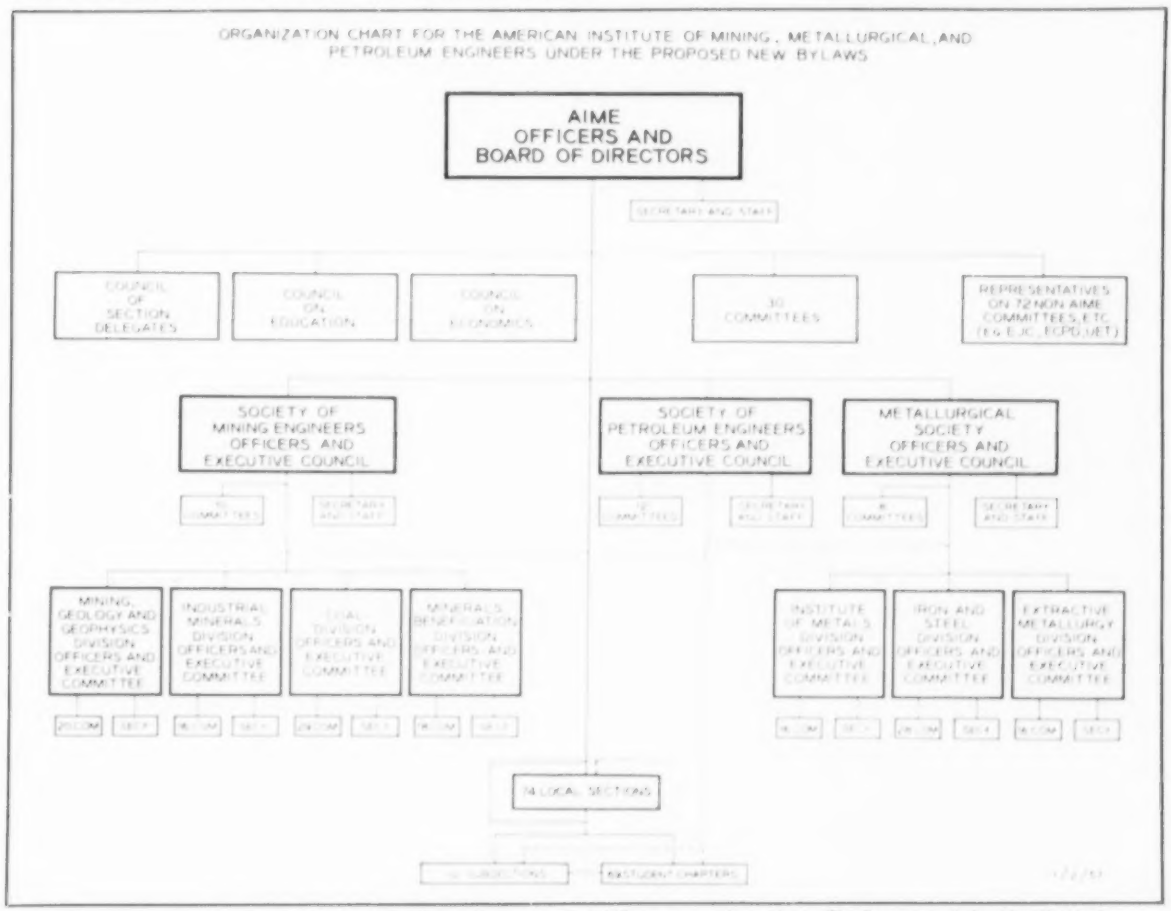
On Jan. 7, the Section met again to hear William D. Manly of the Metallurgical Div., Oak Ridge National Laboratory. Dr. Manly addressed the group on the subject of nuclear metallurgy.

• The **New York Section** was host to AIME President Reistle on Dec. 5, when he took part in a symposium at the Mining Club on *Fuels for Today and Tomorrow*. Coal was discussed by J. R. Forsythe; the uranium phase by W. W. Galbreath.

The New York Section Executive Committee, at its meeting Nov. 13, acted to make $3,000 available as a four-year scholarship to undergraduate students pursuing courses in mining, metallurgy, geology or petroleum. The scholarship will be administered by the Woman's Auxiliary of AIME. This resolution was unanimously approved at the Section meeting on Dec. 5. The money for the award will be taken from funds available to the New York Section, without disturbing the present balance in the reserve fund accounts.

• The **St. Louis Section's** dinner-meeting Jan. 11, in the Hotel York, featured a talk by R. W. Geehan, chief, mineral technology division, USBM, Rolla, Mo., who offered *An Appraisal of Russian Engineers and their Equipment*. Mr. Geehan visited Leningrad in 1956 for meetings of the International Standards Organization's Technical Committee 65, (manganese ore) and also saw mining equipment exhibits in Moscow.

ORGANIZATION CHART FOR THE AMERICAN INSTITUTE OF MINING, METALLURGICAL, AND PETROLEUM ENGINEERS UNDER THE PROPOSED NEW BYLAWS


AIME
OFFICERS AND
BOARD OF DIRECTORS
SECRETARY AND STAFF
COUNCIL OF SECTION DELEGATES
COUNCIL ON EDUCATION
COUNCIL ON ECONOMICS
30 COMMITTEES
REPRESENTATIVES ON 72 NON-AIME COMMITTEES, ETC. (E.G. EJC, EPD, IJET)
SOCIETY OF MINING ENGINEERS OFFICERS AND EXECUTIVE COUNCIL
SOCIETY OF PETROLEUM ENGINEERS OFFICERS AND EXECUTIVE COUNCIL
METALLURGICAL SOCIETY OFFICERS AND EXECUTIVE COUNCIL
15 COMMITTEES
SECRETARY AND STAFF
12 COMMITTEES
SECRETARY AND STAFF
8 COMMITTEES
SECRETARY AND STAFF
MINING, GEOLOGY AND GEOPHYSICS DIVISION OFFICERS AND EXECUTIVE COMMITTEE
INDUSTRIAL MINERALS DIVISION OFFICERS AND EXECUTIVE COMMITTEE
COAL DIVISION OFFICERS AND EXECUTIVE COMMITTEE
MINERALS BENEFICIATION DIVISION OFFICERS AND EXECUTIVE COMMITTEE
INSTITUTE OF METALS DIVISION OFFICERS AND EXECUTIVE COMMITTEE
IRON AND STEEL DIVISION OFFICERS AND EXECUTIVE COMMITTEE
EXTRACTIVE METALLURGY DIVISION OFFICERS AND EXECUTIVE COMMITTEE
14 LOCAL SECTIONS
16 JUNIOR SECTIONS
EXTENSION CHAPTERS

Divisional secretaries for the Society of Mining Engineers of AIME are nonstaff members. The Secretary of the Metallurgical Division secretaries for the Society of Mining Engineers of A of its Divisions and some of its committees.

# Institute Honors E. H. Robie

*Edward Hodges Robie*

WHEREAS, the Board of Directors of the American Institute of Mining, Metallurgical, and Petroleum Engineers takes note that the Secretary Emeritus of the Institute, EDWARD HODGES ROBIE, will retire November thirtieth, Nineteen hundred and fifty-six, after twenty-five years of devoted service,

RESOLVED, that the Board of Directors commends Edward Hodges Robie for his outstanding ability as Editor of *Mining and Metallurgy* from 1932 through 1948, and for his inimitable style combining wisdom and wit as author of the "Drift of Things;" and

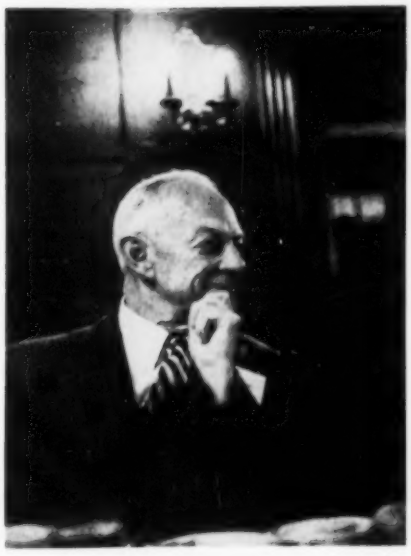
Expresses its sincere appreciation to him for successfully guiding the Institute as Secretary from 1948 to 1955 through a period of far-reaching development and growth.

November 16, 1956 — C. E. Reistle, Jr., President

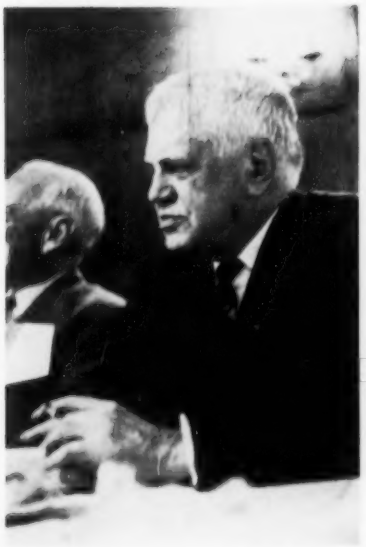
On December 5 the New York Section meeting was the occasion of a salute to Edward H. Robie and his years of service to the Institute. President C. E. Reistle, Jr., on behalf of the Directors, presented Mr. Robie with a *hi-fi* set in token of their personal appreciation and esteem. These candid pictures taken during the meeting show some of the members present with Mr. Robie on this occasion.

*President C. E. Reistle, Jr.*

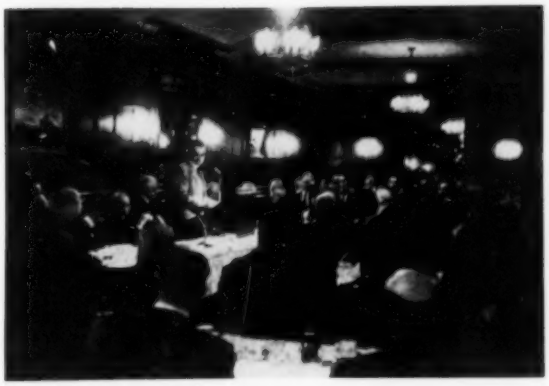
*H. DeWitt Smith and Mr. Robie*



*E. H. Robie*



*Col. H. G. Moulton*



*H. Z. Stuart*
*New York Section Chairman*

# Electric Furnace Steel Conference

Head table guests at the Fellowship Dinner, left to right, J. T. Richey, chairman, Local Arrangements Committee; J. R. Atkinson, president, Electric Metal Makers Guild; H. N. Barrett, Jr., chairman, Finance Committee; E. Kirkendall, AIME Secretary; C. F. Staley, past chairman, Executive Committee; C. E. Simms, chairman, Executive Committee; E. R. Johnson, vice president, Republic Steel Corp.; and E. C. Logelin, Jr., vice president, U. S. Steel Corp., toastmaster.

E. R. Johnson addressed his audience on the interesting subject of *Men, Money, and Machines* at the Fellowship Dinner which was held on Dec. 7 at the Morrison Hotel.

In Chicago, from the 5th through the 7th of December, the Morrison Hotel was the scene of the Fourteenth Annual Electric Furnace Steel Conference. Sponsored by the Electric Furnace Steel Committee of AIME Iron and Steel Div., the three-day conclave chalked up a record attendance of 979, including participants from Canada, Mexico, and the countries of Western Europe.

Wednesday featured plant trips to South Chicago Works of Republic Steel Corp., and to Indiana Harbor Works, American Steel Foundries. Host companies provided conference members with a tasty lunch of South Chicago Fried Chicken and Sautée of Lake Fish. The American Steel Foundries visit showed electric furnace operation applied to foundry practice, and at Republic, members toured the world's largest electric furnace shop.

At the Annual Dinner, Conference Chairman, D. L. Clark, presented the achievement award and a 400-day clock as appreciation by members of the Electric Furnace Steel Committee to outgoing Chairman C. E. Sims. A lively spirit was maintained by toastmaster D. C. Logelin, Jr., vice

A tour of the Indiana Harbor Works of American Steel Foundries took place on the opening day of the conference. Right, gathering for inspection of the plant, left, participants are shown viewing details of furnace operation.

# Breaks All-Time Attendance Records

Head table guests at the Fellowship Dinner, left to right, AIME President C. E. Reistle, Jr.; D. L. Clark, chairman, Conference Committee; R. R. Jones, works manager, American Steel Foundries; C. P. Cutler, district manager, Republic Steel Corp.; V. J. Nolan, chairman, Membership Committee; V. E. Zang, vice chairman, Conference Committee; R. W. Shearman, AIME Metals Branch Secretary.

president, U. S. Steel Corp. AIME President Carl E. Reistle, Jr., had words of greetings to the conference. *Men, Money, and Machines* was the interesting theme of the address by E. R. Johnson, vice president—operations, Republic Steel Corp.

Some 30 papers were presented. *A Broad View of Electric Furnace Steelmaking* was the initial conference session. Robert Boutigny of Stein et Roubaix, emphasized the extensive use of electric furnaces by the Italian steel industry in his review of European practice. Samuel Arnold, 3rd., drew attention to highlights in the history of electric furnace operation, and a paper by Robert Bry of General Electric Co., emphasized the place of transformers in past, present, and future developments. Chapin Hoskins of the Institute of Trend Research, pointed out the most remarkable cyclical movement of scrap prices, a topic of great value to those concerned with supplying electric furnaces.

Other sessions of the conference were devoted to solidification of electric furnace steels, quality control, hot tearing, ladle refractories, use of oxygen, and fume control.

The session on scrap resources seemed of particular interest in view of the present high price of scrap which has led to a search for substitute charging materials. Use of molten iron, desiliconized iron, and sponge iron was considered. John Stalhed of Stora Kopparberg told of the use of sponge iron at his firm's plant in Sweden.

Participants left the conference with a feeling that a great deal had been gained.

## Memorial Award Dinner Honors Frank J. Tone

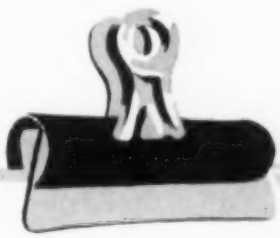
The first annual Frank J. Tone Memorial Award Dinner of the Niagara Frontier Section was held Nov. 28, in the Hotel Niagara. The scroll and medal, presented posthumously to Frank J. Tone, was accepted by F. Jerome Tone, in his father's name. The award was endowed by the late Mr. Tone's two sons, F. Jerome and Franchot Tone.

Prior to the ceremonies, Harry C. Martin, vice president, Carborundum Co., reviewed the winner's life and contributions to science. Mr. Tone had been president of Carborundum Co. Other speakers included AIME President Kinzel, Dr. E. O. Kirkendall, Secretary of the Institute, and Charles W. Hart, section chairman, who acted as toastmaster.

## To Address Institute

*(Continued from page 293)*

War Shipping Administration. He returned in 1944, as a director, and resumed his management of Socony's transportation activities. That same year he was made president and chairman of the executive committee; retaining the latter post, he became chairman of the board in 1955.

# Ready to Serve

. . . those industries and companies which have set their sights on the profitable possibilities in the nuclear chemistry field, the Davison Chemical Co. has consolidated its operations in THORIUM AND RARE EARTHS CHEMICALS.

Two plants, one at Pompton Plains, N. J. (formerly Rare Earths, Inc.) and one at Baltimore, Md. are in operation. Your inquiries concerning our products and services are welcomed.

**DAVISON CHEMICAL COMPANY**

*Division of W. R. Grace & Co.*

**Baltimore 3, Md. and Box 488 Pompton Plains, N. J.**

# Award Winners To Be Honored At Annual Meeting

## Mathewson Medal

**Paul Gordon**, winner of the Mathewson Gold Medal, was born in Hartford, Conn. in 1918. He received a B.S. degree in metallurgy in 1939 and later an M.S. and Ph.D. from the Massachusetts Institute of Technology. Dr. Gordon taught metallurgy at MIT from 1940-1943, during which period he did research on the science of transformation in high speed steels. He was group leader in charge of physical metallurgy for MIT's Manhattan Project, which studied the metallurgical behavior of uranium, beryllium and their alloys, particularly their use in nuclear applications.

From 1949 to 1951, Dr. Gordon was Asst. Prof. at Illinois Institute of Technology. He started his calorimetric investigations of solid state phenomena in metals at the University of Chicago, from 1951 to 1954. Part of this research resulted in the paper for which he has been awarded the Mathewson Medal, *Microcalorimetric Investigation of Recrystallization of Copper*, September 1955 JOURNAL OF METALS. In September 1954, Dr. Gordon was appointed Assoc. Prof., Illinois Institute of Technology, Dept. of Metallurgical Engineering. In addition to his teaching duties, he acts as metallurgical consultant to several nearby industrial firms, and has been active in the guidance of student research. A member of AIME since 1943, Dr. Gordon is active on both a local and national scale.

## Robert W. Hunt Award

The winner of the Robert W. Hunt Medal is Arthur Tix, a native of Germany. Born in 1897, he received an engineering degree in 1922.

He worked as a rolling mill assistant, at Bochumer Verein, eventually becoming manager of the rolling mills and refining departments, steel industry section. During World War II, Dr. Tix became Chairman of the Board of Hanomag, Hanover.

In 1947, Dr. Tix was a director of Stahlwerk Osnabrück, and in 1950 returned to Gusstahlwerk Bochumer Verein. Here, he succeeded in rehabilitating a heavily damaged war plant, and simultaneously developed the steel degassing process in vacuum, which is having far-reaching effects. Dr. Tix is currently Works Director and member of the Board of Gusstahlwerk Bochumer Verein at Bochum, Germany.

The silver medal is in recognition of his paper, *Production-Scale Vacuum Steel Degassing*. JOURNAL OF METALS, April 1956.

D. W. FUERSTENAU

## Receives Hardy Medal

The Robert Lansing Hardy Gold Medal has been awarded to D. W. Fuerstenau of the Niagara Frontier Section. Morris Cohen, Chairman of the 1956 Award Committee, announced at the Board of Directors Meeting in December that Mr. Fuerstenau was the first recipient of this award, with the approval of the Metals Branch Council.

The presentation took place on Feb. 6, in the Hotel Niagara. After dinner Dr. Fuerstenau spoke on *Metal Extraction by the Virgins of Ancient Greece to the Modern Atomic Age*.

A research engineer in the Metal Research Laboratories, Electro Metallurgical Co., Dr. Fuerstenau received his B.S. in metallurgy from South Dakota School of Mines & Technology. Later he obtained an M.S. from Montana School of Mines, and a doctorate in mineral engineering at Massachusetts Institute of Technology. He was Associate Professor at MIT for three years prior to joining Electromet in 1956.

The award, established by Arthur C. Hardy of MIT, as a memorial to his son, recognizes exceptional promise in the field of metallurgy, and the recipient must be under 30 years of age.

## EMD Award

The EMD Award, established in 1955 for the best paper in the field of extractive metallurgy, will be presented this year to **Raymond C. Bell, Gordon H. Turner** and **Ernest Peters** for their paper, *Fuming of Zinc from Lead Blast Furnace Slag—A Thermodynamic Study*. JOURNAL OF METALS, March 1955.

A graduate of the University of British Columbia, Mr. Bell received a B.A. and a B.A.Sc. in chemical engineering in 1938. He worked for The Consolidated Mining & Smelting Co. of Canada, and later was transferred to their Central Research Laboratory where he was promoted from assistant to senior research engineer. During 1949, Mr. Bell did research for Cominco at the Mines Branch of the Dominion Government, Ottawa.

Mr. Turner was born in Vancouver, B. C., and majored in physical chemistry at the University of British Columbia where he also received an M.A. degree in 1941. He joined Cominco at Trail, B. C. in 1944, after service with the Dept. of Mines, British Columbia. Mr. Turner, who has been associated with the research and development division of Cominco, was appointed senior research engineer in 1956.

Ernest Peters was born in Manitoba, Canada in 1926. After serving with the RAF from 1943 to 1945, he returned to school and received the degrees of Bachelor and Master of Applied Science in metallurgical engineering from the University of British Columbia. In 1956, the degree of Ph.D. in metal chemistry was conferred on him. From 1949 to 1950, Dr. Peters served as process control observer with the Geneva Steel Co. From 1951 to 1953 he was junior research engineer for Cominco. He also taught metallurgy at the University of British Columbia. Dr. Peters is currently research engineer, Metals Research Laboratories, Electro Metallurgical Co., Niagara Falls, N. Y.

## Wins Fairless Award

**Leo F. Reinartz,** recipient of the Benjamin F. Fairless Award, was born in 1888 in E. Liverpool, Ohio. He graduated from Carnegie Institute of Technology which also gave him the Alumni Award in 1950. A technical scholarship was established there in his honor in 1954 by the AIME, NOHC. Beginning as chemist at the American Rolling Mill Co., he advanced to Asst. Supt. and later Supt. of the East Side and Central Works Open Hearth Depts. In 1951 he became vice president, Special Operating Development, a post he held until his retirement in October 1956. At present, he is consultant to the president of Armco.

An active AIME member since 1925, Mr. Reinartz served as Director for many terms and was President in 1954. His citation reads: "For distinguished achievement in production and technology. For unerring guidance to the beginning metallurgist, invaluable advice to the operator. For inspiration to both."

*(Continued on page 305)*

## World Metals Congress

The Second World Metallurgical Congress will be held in Chicago, Nov. 2-8, 1957, simultaneously with the 39th National Metal Congress and Exposition. Both events are sponsored by ASM. More than 500 metal scientists, from over 35 nations of the free world, are expected to attend.

The World Congress will be preceded by two weeks of conducted tours during which important plants in the metalworking industry will be visited. The tours will be arranged to cover various fields of the metals industry in nine separate groups, as follows: steelmaking and refining; nonferrous refining and fabrication; ferrous fabrication; heat treatment; welding and joining; inspection and testing; management problems in the metal industry; research (private, industry, government, university); and metallurgical aspects of atomic energy.

The First World Metallurgical Congress was held in Detroit, in 1951, when 510 conferees from 39 free nations of the world participated in tours and meetings.

Dr. Zay Jeffries, world-renowned author and scientist, director general of the first congress and a former president of ASM, has been appointed director general of this Second World Metallurgical Congress.

For information write to American Society for Metals, 7301 Euclid Ave., Cleveland 3, Ohio.

## Metal Exposition And Congress in Los Angeles

Thirty-six national technical societies, including AIME, are cooperating with ASM in presenting the 10th Western Metal Exposition and Congress, March 25-29 in Los Angeles.

Among the feature programs to be presented by the Metals Branch of AIME in the Ambassador Hotel are sessions on *Alloys for Future Airframes, Ultra High Temperature Materials*, and *Light Alloys*, the latter chaired by Harold Block of Ai-Research Corp., Los Angeles.

Other sessions scheduled will deal with corrosion and metal protection, and metallurgy of electronics. A series of conferences will also be devoted to titanium.

The exposition, filling the Pan-Pacific Auditorium and four enormous pavilions, will show how to increase production, cut expenditures, and expedite output.

## Western NOHC Meeting

The AIME NOHC Western Section held a dinner meeting on January 16, at the Rodger Young Auditorium in Los Angeles. Featured speaker was C. A. Rodgers, who presented a paper entitled *A Resume of Oxygen Steel Production and Future Production Facilities at Kaiser Steel Co.*

# ENGINEER*

*"EJC's Report on What's Happening in Engineering"*

*A Periodic Newsletter from Engineers Joint Council • 29 W. 39th Street, New York 18, N. Y.

*(Contents prepared by Engineers Joint Council. AIME is a member.)*

**Survey of Engineering Profession**

EJC Board and ECPD Council are proceeding to establish an operating committee for largest survey of engineering profession ever undertaken. To penetrate every aspect and level of profession, survey will take two years. Recommendations of Task Committee on Survey, point to immediacy of many critical problems, and that size and shape of future national product, the face of city and countryside and even political power at world conference tables will depend on future pattern of engineering. It's that important.

**Busy B's (Election Extra)**

Joseph W. Barker, ASME, and Fischer S. Black, AIEE, unanimously elected president and vice president of EJC for 1957 at Board meeting, No. 16. E. P. Lange, secretary, E. L. Chandler, treasurer, and L. K. Wheelock, assistant secretary, continue. Installation Jan. 18 at close of General Assembly.

**EJC Third Annual Assembly**

National forum for discussion of engineering professionalism and its relation to society. Topics: Place of the engineer nationally in defense on international technological *cold front*, and his income status. Speakers: Henry T. Heald, president, Ford Foundation; Maj. Gen. J. B. Medaris, and Wernher Von Braun, U. S. Army Ballistic Missile Agency; Prof. John Bell Rae, Case Institute of Technology; W. A. B. Iliff, vice president, World Bank; Philip Young, Commissioner, U. S. Civil Service Commission; C. W. Flesher, International Cooperation Administration; J. T. Duce, vice president, Arabian American Oil Co.; C. P. Dunn, president, International Engineering Co.; E. B. Peck, formerly Esso Research & Engineering Co.; D. R. Krotz, California Research Corp.; Frank Leamer, Bell Labs Inc. Dates: Jan. 17-18, with Assembly Dinner evening of 17th. Place: Hotel Statler, N. Y.

**Engineering and Science Closer**

Engineering liaison with science firmer as a result of appointment of E. P. Lange as secretary, Section M, Engineering AAAS. This is a four-year working agreement, not honorary appointment.

**1957 Nuclear Congress**

Looks like biggest and best. Comprises EJC's Nuclear Engineering and Science Conference; NICB's Atomic Energy in Industry Conference; International Atomic Exposition; Hot Labs and Equipment Conference. 60 papers on handling radioactive materials and hot cell operations; 130 papers on fusion, safety, standardization, protection of water supplies, high intensity radiation processing, use of educational reactors, reactor design, etc. Discussions on developing products through use of isotopes, market potentials in atomic energy, legislation, prospects on economic nuclear power and new products through radiation chemistry, are a few of the subjects covered in the meetings. Many new firms exhibiting. The time: March 11-15, 1957. The place: Convention Hall, Philadelphia. Information at EJC, 29 West 39th St., New York.

**Add Labor Panel**

Judge Burnita Mathews, U. S. District Court, D. C., has ruled against the National Labor Relations Board attempt to lump non-professionals and pros as bargaining group, upholding EJC's brief as "friend of Court" and EJC's position as written into the Taft-Hartley Act.

**Help Wanted**

ASEE has appealed to EJC for help in stemming exodus of teachers from schools to industry, confirming EJC-EMC Faculty Study (copies available at EJC, 29 W. 39th St. N.Y.C.) Suggestions to correct situation wanted by ASEE Sec. W. L. Collins, University of Illinois, Urbana, Ill.

## Famous Swedish Scientist Addresses AIME Meeting

The AIME Physical Metallurgy Group, New York Section, was honored at their meeting on Jan. 9, by the presence of Carl Borelius, Professor of Physics, Royal Institute of Technology, Stockholm, Sweden.

An afternoon visit to Philips Laboratories Inc., a division of North American Philips Co., at Irvington-on-Hudson, included a conducted tour of laboratories for magnetics, solid state physics, metallography, air liquefaction, and semiconductors. This was followed by a dinner-meeting in the Harmony Restaurant in Dobbs Ferry. As featured speaker, Dr. Borelius chose to discuss *The Early Stages of Precipitation—Alloys*.

Interested mainly in the physics of metals, Dr. Borelius was chosen IMD Annual Lecturer in 1951 at which time he spoke on *Kinetics of Precipitation in Supercooled Metallic Solutions*. He will spend a month this year at the University of Illinois lecturing on metallurgy.

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## 1957 Nuclear Congress

Peacetime uses of atomic energy will be the topic of the 1957 Nuclear Congress, scheduled for March 11-15 at Philadelphia's Convention Hall. Coordinated by Engineers Joint Council, the session will bring together leaders in science, engineering, industry, government, education, agriculture and other fields.

Included in the Congress are four major elements, including the Second Nuclear Engineering and Science Congress, sponsored by 20 engineering and scientific societies. During the five-day program 130 technical papers will be presented dealing with various phases of nuclear operations, from mining to the disposal of radioactive waste. Emphasis will be on new developments of potential value to civilian industry, especially in the fields of metallurgy, chemical processing, mechanical and power application.

The Fifth Hot Laboratories and Equipment Conference, sponsored by the Hot Laboratories Committee, Oak Ridge National Laboratory, Tenn., on March 14-15th, will deal with operation and development of equipment for atomic energy labs.

Walter G. Whitman of Massachusetts Institute of Technology, is general chairman for the 1957 Congress. President of the AIChE, he was also secretary-general of the U. N. *Atoms For Peace* Conference in Geneva, 1955. According to Dr. Whitman, "The 1957 Nuclear Congress will continue the remarkable cooperation among engineers and scientists from almost every portion of the globe and from every branch of science and engineering for the benefit of mankind, that was first evidenced at Geneva and later at the first EJC Congress in Cleveland in 1955. Progress in the civilian uses of atomic energy is being immeasurably expedited through this cooperative action."

Topics which will be covered by the Fifth Atomic Energy in Industry Conference are: fusion; recent developments in radiation chemistry and radio-isotopes applications; foreign and domestic markets for atomic energy products; nuclear propulsion; waste disposal; health and safety problems; the economics of new reactor types; the latest developments in atomic energy legislation; and problems in local and state atomic energy regulations.

The 1957 Congress expects representatives from other nations to participate in the meetings and to exhibit at the Exposition.

In addition to the four major organizations, the following partial list of organizations will be represented at the 1957 Congress: AIME, ASCE, ASME, AIEE, AIChE, ACS, ASM, IRE, SAE, ASTM and the American Waterworks Assn.

## Institute Awards

*(Continued from page 302)*

### J. E. Johnson Award

The J. E. Johnson, Jr. award will be presented to Raymond W. Sundquist. "For his ability as a Blast Furnace Operator in assisting in the development of new techniques and his contribution to the Blast Furnace literature."

Born in St. Paul, Minn. in 1916, he received the degree of metallurgical engineer from the University of Minnesota in 1938. During the summer of 1937, he worked as a rodman at the South Chicago Works of U. S. Steel Corp. After graduation, the same company employed him at their Gary Works as a practice apprentice in the Blast Furnace Dept. Mr. Sundquist advanced steadily. In 1954 he became Division Superintendent of Blast Furnaces.

### Douglas Gold Medal

Russel B. Caples has been chosen to receive the James Douglas Gold Medal. Born in Glasgow, Mo., in 1888, he received a B.S. degree in mining engineering in 1910, and in 1948, an honorary Ph.D. in engineering from Missouri School of Mines.

Mr. Caples worked in the testing and research departments, Anaconda Reduction Works, until 1914. During this period he collaborated in developing the electrolytic zinc process. In 1953 he was transferred to the New York office of The Anaconda Co. as president of Anaconda Aluminum Co., and in 1955 was made vice president-metallurgical operations, of all the Anaconda Co.'s properties.

A director of many organizations including AIME, Mr. Caples' citation reads: "For distinguished achievement in extractive metallurgy, particularly in the electrowinning of zinc; and for outstanding success in stimulating the professional careers of many metallurgists."

## Mortada Wins Two Prizes

Two of the Institute's most coveted awards will soon grace the home and the purse of young Mohamed Mortada, author of the prize winning paper, *A Practical Method for Treating Oilfield Interference in Water-Drive Reservoirs*. The Rossiter W. Raymond Award consists of a $100 cash prize and a certificate. A $400 cash award and a certificate comprise the Alfred Noble Award. Both awards will go to Mr. Mortada for his paper, the first he ever submitted, which appeared in JOURNAL OF PETROLEUM TECHNOLOGY, December 1955.

Mr. Mortada's preparation for this fruitful investigation was begun at the University of Cairo in his native Egypt, where he earned a chemical engineering degree. He entered the University of California in 1947, to take both his M.S. and Ph.D. degrees in petroleum engineering.


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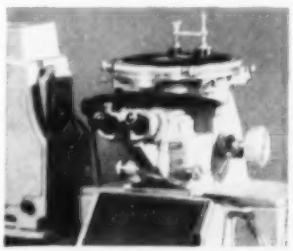

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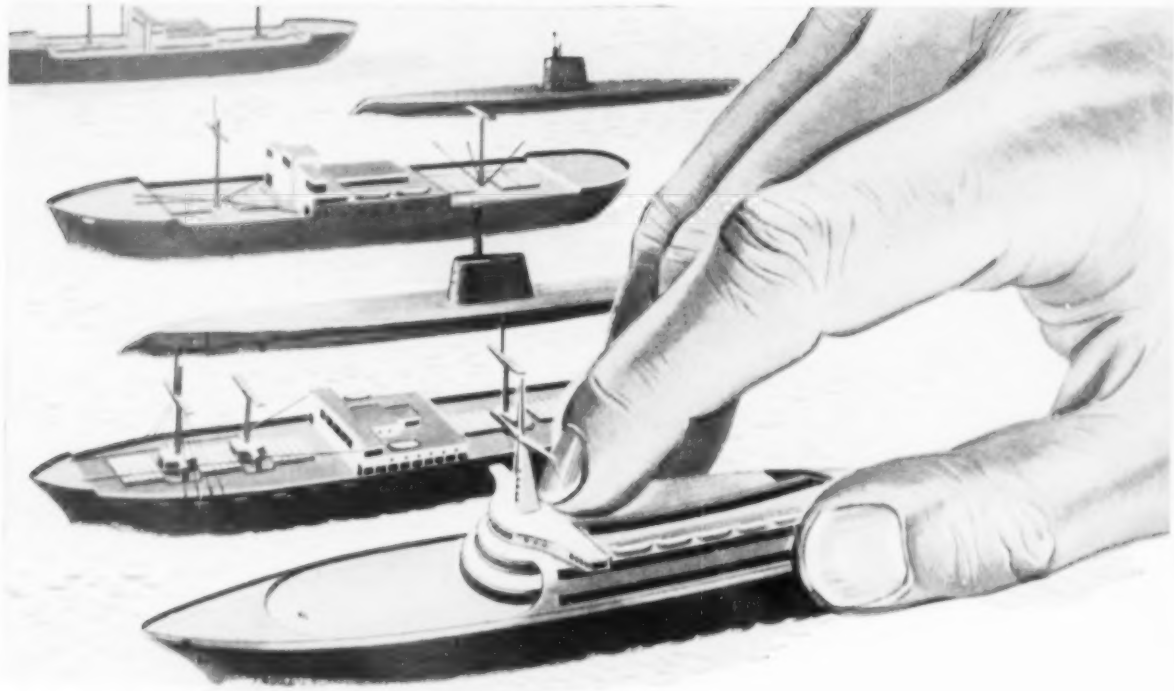

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## PERSONALS

W. ROSTOKER

**William Rostoker** has been promoted to assistant manager, metals research department, Armour Research Foundation, Illinois Institute of Technology, Chicago. He had previously served as senior metallurgist and supervisor of physical metallurgy. Prior to joining the foundation in 1951, he taught at Illinois Institute of Technology and the University of Birmingham, Eng. A native of Toronto, Dr. Rostoker received his B.S. and M.S. degrees in metallurgy from the University of Toronto, Canada, and his Ph.D. from Lehigh University, Bethlehem.

**Clarence Zener** has been named director of the research laboratories, Westinghouse Electric Corp., Pittsburgh. Formerly a professor and physicist at the University of Chicago, Dr. Zener had been associate director for the past five years. A graduate of Stanford University, Palo Alto, Calif., he earned his Ph.D. degree in physics at Harvard University. Upon his return from postdoctoral study at the University of Leipzig, Ger., Dr. Zener received a National Research Fellowship in physics. He has taught at Washington University, St. Louis; City College, N. Y.; and was associate professor at Washington State College. A leading researcher in physical metallurgy, Dr. Zener's work at Westinghouse has led to the development of a durable new alloy, Nivco.

**Harold E. Lee** has been appointed vice president in charge of research and development at The Bunker Hill Co., San Francisco. On the staff since 1931, he had been manager of metallurgy, in charge of ore procurement and responsible for the company's reduction plants.

**S. A. Rosecrans** is manager, Materials Engineering Dept., Westinghouse Electric Corp., East Pittsburgh.

**James W. Snyder, Jr.,** has joined Aluminum Co. of America, Buffalo, as assistant metallurgist.


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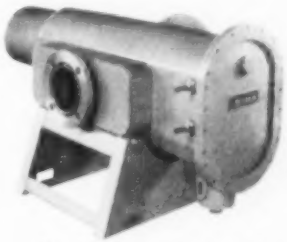

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Titanium Metals Corp. of America has recently announced the following appointments to the Technical Dept., Metallurgical Research Div. Staff, Henderson, Nev.: **M. L. Greenlee, A. S. Gahunia, A. A. Hatch,** and **J. V. Luhan.**

A graduate of the Colorado School of Mines, Mr. Greenlee is a member of the ASM. Mr. Gahunia, who attended the University of Punjab, India, received his B.S. degree in metallurgical engineering from the University of Washington, Seattle. Mr. Hatch, a graduate of Ohio State University, was formerly research metallurgist for Clevite Research Center, Cleveland, and Menasco Manufacturing Co., Burbank, Calif. Mr. Luhan has worked for the past two years as research assistant, Case Institute of Technology, where he received his B.S. in metallurgical engineering and M.S. in physical metallurgy.

**Leslie L. Seigle** has recently been appointed manager of metallurgy research, Stet Research Laboratories, Sylvania Electric Products Inc., Bayside, N. Y. He had been directing basic metallurgy programs, particularly the coordination of non-nuclear metallurgy research. A chemical engineering graduate of Cooper Union, Dr. Seigle received his master's degree in metallurgy from the University of Pennsylvania in 1949 and his Ph.D. in 1951 from MIT. After serving as research metallurgist with International Nickel Co., and project leader at the Thermodynamics Laboratory, University of Pennsylvania, Dr. Seigle joined Sylvania in 1951. He was appointed adjunct professor of metallurgy at New York University in 1952.

**H. Edward Fuhrmeister** has left Henderson, Nev., for Brackenridge, Pa., and a job in the titanium section of Allegheny Ludlum Steel Corp.

Allegheny Ludlum Steel Corp. has announced the appointment of **S. Ramachandran** and **H. L. Bishop, Jr.,** as advisors in the Research Laboratory, Brackenridge, Pa. Both men were associated with Youngstown Sheet & Tube Co., and attended Massachusetts Institute of Technology where each received the degree of doctor of science.

**Malcolm F. Judkins,** manager, New Products Div., Firth Sterling Inc., addressed the Purchasing Agents Association of Indianapolis on the subject of power reactors.

**Roger W. Straus** and **Benjamin F. Fairless** are co-chairmen of The National Conference of Christians and Jews, which sponsored Brotherhood Week, Feb. 17-24. Mr. Straus is one of the founders of the Conference, begun in 1928, to promote justice, understanding, and cooperation among all faiths and to eliminate intergroup prejudices.

**W. C. McCosh** has been appointed manager of railroad and industrial brush sales for National Carbon Co., a division of Union Carbide & Carbon Corp., with offices in New York.

**Paul Albert** was named supervising engineer, heading the materials engineering group in soft magnetic alloys, at Westinghouse Electric Corp., Pittsburgh.

**John E. Stukel** has joined the staff of Cleveland Cliffs Iron Co. He had been with Youngstown Sheet & Tube Co., Youngstown, as a development engineer.

**Harry W. Weart** has completed his studies at the University of Wisconsin and is now research engineer at the Westinghouse Research Laboratory, Pittsburgh.

**Donald LaVelle,** former assistant manager, Aluminum Dept., American Smelting & Refining Co., Barber, N. J., is now with Kaiser Aluminum & Chemical Sales Inc., Chicago, as assistant product manager for pig, ingot and billet.

## MEMBERSHIP

*Proposed for Membership*
*Metals Branch, AIME*

*Total AIME membership on Dec. 31, 1956, was 26,298; in addition 3,415 Student Associates were enrolled.*

*ADMISSIONS COMMITTEE*

*R. B. Caples, Chairman; F. A. Ayer, Vice-Chairman; A. C. Brinker, R. H. Dickson, C. R. Dodson, R. B. Fulton, T. D. Jones, F. W. Hanson, Sidney Rolle, F. T. Sisco, O. B. J. Fraser, F. W. McQuiston, Jr., A. R. Lytle, L. P. Warriner.*

*The Institute desires to extend its privileges to every person to whom it can be of service, but does not desire as members persons who are unqualified. Institute members are urged to review this list as soon as possible and immediately to inform the Secretary's office if names of people are found who are known to be unqualified for AIME membership.*

**Members**

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Alfred P. Bissacca, Los Angeles
James H. Bischoff, Coppersburg, Pa.
Paul P. Combs, Trenton, Mich.
Terence E. Daney, Library, Pa.
Harold L. Farling, Cleveland
Hugh S. Graham, Bethlehem
Arthur F. Gross, Springfield, Ohio
N. W. Hendry, Asbestos, Que., Canada
C. E. Judson, Lachine, Que., Canada
W. E. Keffer, Copperhill, Tenn.
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Harvey W. Maurice, Pittsburgh
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Douglas Grant McCullough, Rochester, Mich.
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McBeth Sample, Middletown, Ohio
A. Sevilla, Sonora, Mexico
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Ernest Underwood, East Chicago, Ind.
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Robert G. Brown, Parma, Ohio
Homer B. Greetham, Sheffield Lake, Ohio
A. M. Hattingh, Daggafontein Transvaal, South Africa
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R. B. Russell, Glenshaw, Pa.
Thomas I. Sigler, Lincoln Park, Mich.

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Walter H. Bade, Sandusky, Ohio
Thomas E. Barker, Sharon, Pa.
Alan R. Burkart, Cleveland
W. Barry Collins, Berea, Ohio
William P. Collins, Great Falls, Mont.
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William H. Dickey, Pittsburgh
Harlan W. Diefendorf, Syracuse, N. Y.
Karl Foster, Pittsburgh
John H. Gehrke, Beaver Falls, Pa.
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Roland H. Roe, Valdosta, Ga.
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Allyn Robert Von Neida, New Haven, Conn.
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Choh-Yi Ang, Indianapolis
Edward C. Rudolphy, Park Forest, Ill.

**Junior to Member**

Walston Chubb, Columbus, Ohio
T. M. Kegley, Jr., Oak Ridge, Tenn.
Pascal Levesque, Needham, Mass.
John W. Pugh, Cleveland
Victor F. Zackay, Dearborn

**REINSTATEMENT**

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# OBITUARIES

**John C. Jones** (Member 1937), a native of Port Talbot, Wales, died on Dec. 25, 1955. Born in 1904, he received his B.S. and M.S. degrees from University College of Swansea. He remained at his alma mater until 1930 as an assistant lecturer in metallurgy, and during those academic years wrote several technical papers. Mr. Jones was a research metallurgist at The British Aluminium Co. Ltd. until 1937, when he came to the United States as technical advisor to Arthur Seligman & Co. Inc., N. Y. At the time of his death, he was associated with Aluminum Transatlantic Inc., N. Y.

**Charles F. Ramseyer** (Member 1930), president of Ramseyer & Miller Inc., N. Y., died on Dec. 9, 1956. A native of Boston, he was born on Oct. 21, 1900. A graduate of Harvard University and MIT, where he obtained an M.S. degree, Mr. Ramseyer began his career as a research engineer at Illinois Steel Co. In 1931, he joined the Electric Generating Corp., Chicago, where he was employed as chemical engineer until 1949. After an association with H. A. Brassert & Co., he formed the consulting firm which he served as chairman of the board until his death.

**Thomas Z. Humphrey** (Member 1931) died on December 28, 1955, at the age of 65. He was a native of Pomeroy, Wash., and a graduate of Washington State College with a B.Sc. in mining engineering. In 1916, he began a nine-year career with Anaconda Copper Mining Co., interrupted by a tour of duty with the U. S. Army. Mr. Humphrey joined the staff of Andes Copper Mining Co., Potrerillos, Chile in 1925, remaining there as smelter superintendent until 1936. He then accepted the position of furnace refinery superintendent, International Smelting & Refining Co., Perth Amboy, N. J., later transferring to Miami, Ariz., where he was technical consultant.

**Ivor Pounds, Jr.** (Member 1951) died recently. He was born on Feb. 1, 1928, in Montezuma, Kan. and graduated from the Missouri School of Mines & Metallurgy. In 1949 he was employed as a metallurgist, American Smelting & Refining Co., East Helena, Mont., after which he became Lead Smelter Supt., Cerro de Pasco Copper Corp., La Oroya, Peru.

**John G. Sullivan** (Member 1951) died on February 22, 1956. He was born in Pittsburgh in 1914, and received his B.S. in metallurgical engineering from Carnegie Institute of Technology in 1936. Mr. Sullivan began a lifetime career with American Steel & Wire Co. in 1937, broken only by U. S. Navy service from 1943 to 1946. Prior to joining the navy, his work was that of metallurgist, but his post-war career took a supervisory turn. He was appointed superintendent of the Open Hearth Division in 1951.

**Jack Dankowitz** (Member 1951) died recently. Born on Dec. 25, 1900 in Poland, he became a citizen of Bolivia. He was associated with French and South American companies engaged in lead and tin smelting in Argentina, Brazil and Bolivia, working as metallurgist and mill superintendent. Mr. Dankowitz resided in Buffalo Snyder, N. Y.

**Stuart B. Marshall** (Member 1910) who died on Nov. 13, 1956, was a graduate of Virginia Military Institute, class of '94. His first professional assignment took him to Jerome, Ariz., as head assayer for United Verde Copper Co., after which he returned to the East Coast to join the Dunbar Furnace Co., Dunbar Pa. Mr. Marshall left his post as general manager of Dunbar, for a similar position at American Manganese Mfg. Co., and then became general superintendent of Tallassee Power Co., a subsidiary of Aluminum Co. of America, in Badin, N. C. He later established a consulting office in Washington, D. C. Mr. Marshall was a member of the Washington Society of Engineers, and held an honorary M.S. degree from VMI.


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## NECROLOGY

| Date Elected | Name | Date of Death |
|---|---|---|
| 1955 | S. E. Blamey | Sept. 2, 1956 |
| 1937 | C. L. Boeke | Dec. 24, 1955 |
| 1929 | Eugene E. Brossard | Sept. 19, 1956 |
| 1920 | Douglas C. Corney | November 1955 |
| 1951 | Jack Dankowitz | Unknown |
| 1928 | James E. Davidson | Unknown |
| 1914 | Everette DeGolyer Honorary Member | Dec. 14, 1956 |
| 1956 | Victor C. DeMunck | Dec. 12, 1956 |
| 1936 | Ernest V. Faraggi | August 1956 |
| 1956 | E. R. Fritz | Dec. 7, 1956 |
| 1884 | Joseph P. Gazzam Legion of Honor | Unknown |
| 1937 | L. A. Greiling | Mar. 15, 1956 |
| 1947 | George H. Hannum | Aug. 14, 1956 |
| 1894 | J. Gordon Hardy Legion of Honor | Dec. 14, 1956 |
| 1917 | Marden W. Hayward | February 1956 |
| 1951 | Gerardo Heimpel | Aug. 13, 1956 |
| 1953 | John B. Hicks | Nov. 26, 1956 |
| 1954 | Woodrow Knott | Nov. 7, 1956 |
| 1906 | Robert S. Lewis Legion of Honor | February 1956 |
| 1949 | R. I. C. Manning | Dec. 8, 1956 |
| 1928 | Thomas Morch | 1944 |
| 1937 | Ralph Neuhaus | June 16, 1956 |
| 1952 | R. C. Nicholson, Jr. | Oct. 15, 1956 |
| 1940 | Everett Paris | Nov. 3, 1956 |
| 1948 | V. F. Parry | Unknown |
| 1951 | Ivor Pounds, Jr. | Unknown |
| 1930 | Charles F. Ramseyer | Dec. 9, 1956 |
| 1935 | N. P. Rhinehart | Nov. 11, 1956 |
| 1953 | Clarence V. Saylor | Dec. 15, 1956 |
| 1952 | M. I. Signer, Jr. | Nov. 17, 1956 |
| 1948 | William D. Steinle | Unknown |
| 1942 | J. Frank Tindol | November 1956 |
| 1927 | Eugene A. White | Dec. 9, 1956 |
| 1944 | Arnold Ziffzer | Dec. 17, 1956 |

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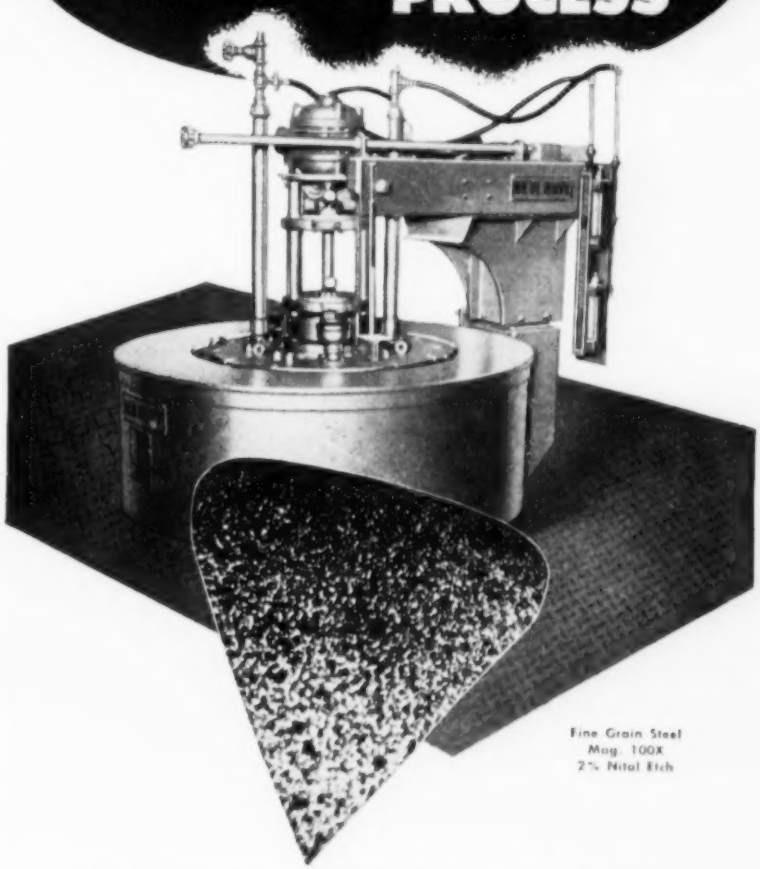

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## COMING EVENTS

**Feb. 8. AIME, NOHC Southwestern Local Section,** annual meeting, Lennox Hotel, St. Louis.

**Feb. 24-28. AIME Annual Meeting,** Roosevelt and Jung Hotels, New Orleans.

**Mar. 10-13. AIChE,** Greenbrier Hotel, White Sulphur Springs, W. Va.

**Mar. 10-16. EIC Second Nuclear Engineering and Science Congress,** Convention Hall, Philadelphia.

**Mar. 11-15. National Assn. of Corrosion Engineers,** annual conference and exposition, Kiel Auditorium, St. Louis.

**Mar. 13. AIME, NOHC Western Local Section,** Hodgen Young Auditorium, Los Angeles.

**Mar. 13. AIME, NOHC Chicago Local Section,** annual all-day meeting, Del Prado Hotel, Chicago.

**Mar. 18-19. AIME Deep Drawing Steel Regional Meeting,** sponsored by authorities on sheet steel, NOHC, and AIME Detroit Section, Rackham Building, Detroit.

**Mar. 25-29. ASM And Others,** Tenth Western Metal Exposition and Congress, Pan-Pacific Auditorium and Ambassador Hotel, Los Angeles.

**Mar. 26. AIME, Connecticut Local Section.** Speaker: P. C. Rossin, Universal-Cyclops Steel Corp.; subject: *Metallurgical Aspects of Vacuum.* Waterbury, Conn.

**April 5, 6. AIME, Pacific Southwest Mineral Industry Conference.** Sponsor: Nevada Local Section; cosponsors: San Francisco and Southern California Local Sections. Reno.

**Apr. 8-10. AIME National Open Hearth Steel and Blast Furnace, Coke Oven, and Raw Materials Conferences,** Penn-Sheraton Hotel, Pittsburgh.

**Apr. 11-13. AIME, Pacific Northwest Regional Conference,** Multnomah Hotel, Portland, Ore.

**Apr. 16-17. AIME Regional Conference on High Temperature Alloys,** Hotel Carter, Cleveland.

**Apr. 22-24. CIM,** annual meeting, Ottawa, Ont., Canada.

**May 5-9. American Ceramic Soc.,** annual meeting, Statler Hotel, Dallas.

**May 6. AIME, NOHC Chicago Local Section,** spring meeting, Phil Smidt's Restaurant, Chicago.

**May 12-16. Electrochemical Soc.,** Statler Hotel, Washington, D. C.

**May 28-29. AIME, Reactive Metals Conference.** Mining Branch sessions on May 28, Metals Branch sessions on May 28, 29. Ambassador Hotel, Los Angeles.

**June 8. AIME, NOHC Southern Ohio Local Section,** annual golf party, Middletown, Ohio.

**June 24-28. ASTM,** annual meeting, Chalfonte-Haddon Hall, Atlantic City, N. J.

**Sept. 8-Oct. 9. Commonwealth Mining and Metallurgical Congress,** British Columbia to Nova Scotia.

**Oct. 6-9. AIME, Petroleum Branch,** Dallas.

**Oct. 9-11. Joint Solid Fuels Conference, ASME-AIME Coal Div.,** Chateau Frontenac, Quebec.

**Oct. 18. AIME, NOHC Southern Ohio Local Section,** annual meeting, Deshler-Hilton Hotel, Columbus, Ohio.

**Oct. 30-Nov. 1. AIME Rocky Mountain Minerals Conference,** Denver.

**Nov. 4-6. AIME, IMD Fall Meeting,** Morrison Hotel, Chicago.

**Nov. 12. AIME, NOHC Buffalo Local Section,** annual meeting, Hamilton, Ont., Canada.

**Dec. 4-6. AIME, 15th Electric Furnace Steel Conference,** Penn-Sheraton Hotel, Pittsburgh.

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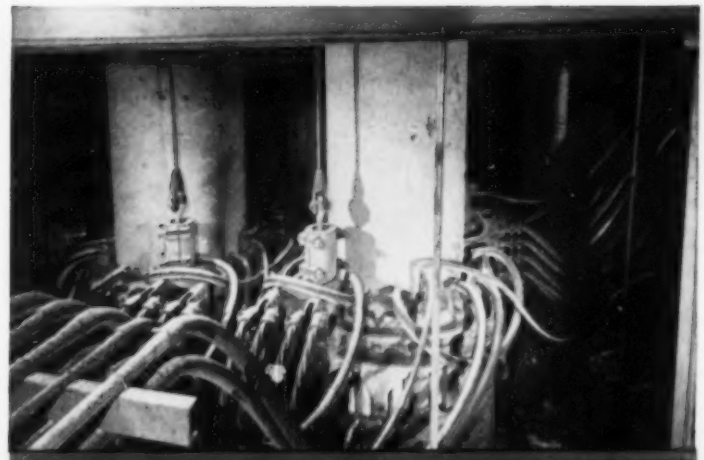

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SILICON, second most plentiful element on earth, is so tightly combined with oxygen that it can be isolated only through complex chemistry and the terrific heat of the electric furnace. Dow Corning are performing this operation with the Lectromelt* furnace equipment shown here, producing 98% pure silicon metal.

In the furnace reaction, quartzite rock is reduced with coke and charcoal at a temperature of 3100°F. Each tapping of the furnace yields about a ton and a half of silicon. Upon cooling, it is ground to a powder and employed in the manufacture of silicones.

Lectromelt engineers, throughout the past 38 years, have been conducting continuing research on high temperature chemistry. They have designed and built many furnaces for various branches of the metallurgical and chemical industries. For their help, write Electrothermic Division, Lectromelt Furnace Company, 326 32nd St., Pittsburgh 30, Penna. (a McGraw Electric Company Division).

● **TOP:** *This Lectromelt Furnace is unique in design. Its suspended roof protects the flexible conductors and water hoses against the intense heat, but leaves the furnace open for manipulation of the charge.*

● **MIDDLE:** *3100° F., silicon's reduction temperature, is maintained at each submerged arc, the electrode moving down automatically as it is consumed. At the right is one of six chutes for charging the furnace.*

● **BOTTOM:** *Smelters, working through six open ports at the charging level, regulate, stoke and trim the flaming mixture of quartzite and coke. The crucible is 13 feet across and almost 7 feet deep.*

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**DATA ON VOLMEX 81 . . .**

# HOW VOLMEX® 81

## performs as a high volatile ingot mold coating when applied . . .

## (1) Wet (2) Dry (3) At varying ingot mold temperatures

VOLMEX 81 is a highly volatile ingot mold coating which has been tested and adopted by a number of steel plants. The charts below are based on actual data obtained during such trial runs. Because of the differing practices employed by various companies, we have included several variables. First, because VOLMEX 81 can be applied either dry or with water as the carrier (spraying, swabbing or dipping are all practical), we have presented test figures on its use, both wet and dry. Second, we have included data on a range of ingot mold temperatures from 100°F. to 700°F., and third, we offer for comparison the results of a one-hour and a three-hour time-at-mold temperatures. No matter what method of application was used, VOLMEX 81 was found to repel metal splashes effectively before they could solidify and cling to the mold wall. At the same time, enough of the graphite-base coating was left at the metal/mold interface to keep the main body of the pour from clinging when it reached the point of splash, thus reducing surface defects. For wet application, VOLMEX 81 is easily stirred into suspension and remains in that state for hours without re-agitation.

VOLMEX 81 is the most volatile of the United States Graphite Company ingot mold washes. Others, in order of their volatility are: 90-B (non-volatile), MEX (very little volatility), and VOLMEX (volatile, but less so than VOLMEX 81). Write us for more extensive details or our engineering staff is available for on-the-spot assistance. Mold spray and mixing equipment are ready for your use in tests you may want to arrange.

**Table I** **Volatility of VOLMEX 81 Ingot Mold Wash**

| Mold Coating Volatility Test—VOLMEX 81 Water Application 2.75 lbs. powder/gal. water—Period—1 Hour at | Temperature 300°F. | 500°F. | 700°F. |
|---|---|---|---|
| % loss after heating for 1 hour | 74.3 | 73.9 | 75.4 |
| % loss on ignition at 1900°F. after 1 hour at specified temperature | 16.1 | 14.4 | 11.3 |
| Total percent volatile matter | 90.4 | 88.3 | 86.7 |

**Table II**

| Mold Coating Volatility Test—VOLMEX 81 Dry Application—Period—1 Hour at | Temperature 100°F. | 300°F. | 500°F. | 700°F. |
|---|---|---|---|---|
| % loss after heating for 1 hour | 0.1 | 0.8 | 0.9 | 2.8 |
| % loss on ignition at 1900°F. after 1 hour at specified temperature | 60.8 | 60.4 | 59.7 | 56.5 |
| Total percent volatile matter | 60.9 | 61.2 | 60.6 | 59.3 |

**Table III**

| Mold Coating Volatility Test—VOLMEX 81 Dry Application—Period—3 Hours at | Temperature 100°F. | 300°F. | 500°F. | 700°F. |
|---|---|---|---|---|
| % loss after heating for 3 hours | 0.1 | 0.8 | 1.1 | 4.5 |
| % loss on ignition at 1900°F. after 3 hours at specified temperature | 60.6 | 59.6 | 59.7 | 53.1 |
| Total percent volatile matter | 60.7 | 60.4 | 60.8 | 57.6 |

You will note in the first chart which is the wet application, that the loss is all of the water contained plus a slight amount of the remaining volatile. Since on a weight basis, the powder is about ¼ of the make-up, if you will multiply the percent loss figures at 1900°F. by four, you will get an idea of the amount of volatile which would be present for loss during the pouring operation.

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